RR

ANALYTICAL ABSTRACTS

TECHNOLOGY DEPT.

Dealing with all branches of Analytical Chemistry: Published Monthly with THE ANALYST by the Society for Analytical Chemistry

PUBLIC LIBRARY
MAY 18 1955
DETROIT

Editor: NORMAN EVERS, B.Sc., Ph.D., F.R.I.C.
20, EASTCHEAP, LONDON, E.C.3
Telephone: MANsion House 6608

Published for the Society by
W. HEFFER & SONS, LTD., CAMBRIDGE, ENGLAND

Volume 2 Price 4s.
Subscription Rate, inclusive of Index, 50s. per annum, Post Free
No. 4, Abstracts 823-1107 April, 1955

Some Important New Serial Works

COMPREHENSIVE INORGANIC CHEMISTRY

Edited by M. Cannon Sneed, J. Lewis Maynard, R. C. Brasted.
To be in 11 vols., of which vol. 1 is now ready. 8vo., 243 pp., 35/- net.

Volume 1 contains Principles of Atomic and Molecular Structure, by W. N. Lipscomb; Theoretical and Applied Nuclear Chemistry, by P. R. O'Connor; The Actinide Series, by G. T. Seaborg.

HANDBUCH DER MIKROCHEMISCHEN METHODEN*

Edited by Fr. Hecht and M. K. Zacherl. To be in 5 volumes.

The first part to appear will be Präparative Mikromethoden in der organischen Chemie, by Hans Lieb and W. Schöniger, with Mikroskopische Methoden, by L. Kofler and A. Kofler.

HANDBUCH DER CHEMISCHEN UNTERSUCHUNG DER TEXTILFASERSTOFFE*

By H. M. Ulrich. To be in 4 vols.

* *A Prospectus is available on application.*

Among important new books are: The Van Nostrand Chemists' Dictionary, 761 pp., **£3.** Physico-Chemical Methods, by Joseph Reilly and W. N. Rae, 5th edition, 2 vols., **£7 10s.** Paper Chromatography, by F. Cramer, 2nd revised and enlarged edition, translated by L. Richards, **25s.** net.

Please order, or ask for further details, from—

W. HEFFER & SONS LTD.

PETTY CURY · CAMBRIDGE · ENGLAND

Telegrams: "HEFFER, CAMBRIDGE." *Telephone:* 58351.

PRICE OF ANALYTICAL ABSTRACTS

Subscription Rate, inclusive of Index .. .. .. 50s. per annum, post free.

Abstracts printed on one side of the page only, exclusive of Index .. .. .. 60s. per annum, post free.

The subscription rate for *The Analyst* together with *Analytical Abstracts* and Indexes is 80s. per annum, post free.

All further enquiries about subscriptions should be made through the Secretary, the Society for Analytical Chemistry, 7-8, Idol Lane, London, E.C.3. Telephone: MANsion House 6608.

Karl Fischer Reagent

NOW REDUCED IN PRICE

Hopkin and Williams Ltd. are pleased to announce the following substantially reduced prices for Karl Fischer Reagent which have been effected as a result of increased production necessitated by the ever-growing demand for this reagent.

The B.T.L. Electrometric Apparatus for Karl Fischer titrations, in use in the laboratories of Hopkin & Williams Ltd.

CODE No. 9350.5

1,000 ml. 30/- 500 ml. 16/-

The reagent is a single solution ready for immediate use and has a water equivalent of 5 mg. of water per ml. The price reduction amounts to approximately 25% but the quality of the reagent and its stability have been maintained at the usual high level.

HOPKIN & WILLIAMS Limited

Manufacturers of pure chemicals for Research and Analysis

FRESHWATER ROAD, CHADWELL HEATH, ESSEX.

RECENT CATALOGUES*

*No. 675: Books on the History of Science and Technology: Antiquarian, Second-hand and New, English and Foreign.
54 pp.

*No. 676: Forthcoming Scientific and Technical Books: a select list.
22 pp.

Catalogues gratis on application

W. HEFFER & SONS
LIMITED
Petty Cury · Cambridge
England

You can make no mistake in employing

WHATMAN
FILTER PAPERS

The makers have perfected a range covering the special requirements of every industry.

- QUALITATIVE GRADES
- EXTRACTION THIMBLES
- SINGLE and ACID WASHED QUALITIES
- HARDENED FILTER PAPERS
- SPECIALITIES

STOCKED BY ALL LABORATORY FURNISHERS

Made by:—
W. & R. BALSTON LTD., MAIDSTONE, KENT.

Sole Sales Agents:—
H. REEVE ANGEL & Co. Ltd., 9, BRIDEWELL PLACE
LONDON, E.C.4.

American Men of Science

A BIOGRAPHICAL DIRECTORY

Ninth Edition — In Three Volumes

Edited by Jaques Cattell

Vol. I. The Physical Sciences. 2180 pages. Imp. 8vo., strongly bound in buckram. £7 10s. 0d.

Vol. II, Biological Sciences, is due this Autumn, and Vol. III, Social Sciences, in the Spring of 1956. Their prices will be announced later; the volumes are expected each to be a little more than half the size of Volume I.

This first volume contains 43,518 entries, each giving a brief account of scientists' appointments and particular lines of work.

Available from

W. HEFFER & SONS LTD.

ENGLISH AND FOREIGN BOOKSELLERS

3-4, PETTY CURY · CAMBRIDGE · ENGLAND

Telephone 58351 — Telegrams: "Heffer, Cambridge"

ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

823. **Energetic characteristics and the analytical classification of ions.** K. B. Yatsimirskii (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 282–292).—The author's theory and scheme of classification (*Brit. Abstr. C*, 1952, 37) are developed further. G. S. SMITH

824. **Application of statistical analysis to analytical data.** P. D. Lark (*Anal. Chem.*, 1954, **26** [11], 1712–1715).—Segregation and estimation of random and systematic errors in an analytical procedure may be accomplished by the principle of least squares, provided that a sufficient range of concn. of the reagents is investigated. The estimates of error are related to the amount of substance under determination, and the subsequent steps of choice of hypothesis for this relationship and computation of regression equations and errors are illustrated by results obtained in the assay of CaO in CaO - MgO mixtures. D. A. PANTONY

825. **Interpretation of data obtained in non-aqueous media.** E. Grunwald (*Anal. Chem.*, 1954, **26** [11], 1696–1701).—Errors arising in titrations in media of low dielectric constant are ascribed to diminution of ionic activities (and hence activity coefficients) due to the formation of ion pairs that cause inaccurate inflexion points in potentiometric methods and salt effects with indicators. The nature of the ion pairs are discussed, and relationships between activity coefficient, dissociation constant and degree of dissociation and concn. are deduced that enable ionic activities to be calculated. Results are applied to the potentiometric titration of HCl - acetic acid mixtures with NaOH in 95 per cent. methanol; methods of improving the inflexion point are suggested. Equations are deduced that enable indicators to be chosen for weak acid - strong base and weak base - strong acid titrations. D. A. PANTONY

826. **The use of complexones in analytical chemistry.** G. Bergshoeff (*Chem. Weekbl.*, 1954, **50** [34–35], 581–589, 593–600).—The general properties, structures and uses of the complexones in analytical chemistry are discussed. The applications dealt with include volumetric analysis, gravimetric analysis, quantitative separations, colorimetry, polarography, use in conjunction with ion-exchangers and qualitative analysis. The literature up to the beginning of 1954 is covered in a bibliography of 202 references. E. HAYES

827. **Current status of analysis by titration.** P. J. Elving (*Anal. Chem.*, 1954, **26** [11], 1676–1679).—Requirements and recent developments of titrimetric analysis are reviewed and are illustrated by reference to the determination of Ca (0·005 to 2 per cent.) in lead alloys. Samples are removed from the molten alloys with a standard ladle and to them are added definite numbers of antimony - lead pellets of standard weight. After being stirred, the samples are cooled and their surface appearances noted; the end-point is marked by the almost complete disappearance of the grey film, a stage corresponding to $\simeq$ 0·005 per cent. of free Ca, the remainder having been removed as Sb_7Ca_9. The Ca concn. is calculated from a calibration curve of percentage of Ca plotted against the number of pellets added. Precision is claimed to be $\pm$ 0·002 per cent. up to 0·11 per cent. of Ca. Other possible titrations in non-liquid media are suggested. D. A. PANTONY

828. **Selection of the medium for a particular titration.** J. S. Fritz (*Anal. Chem.*, 1954, **26** [11], 1701–1704).—Requirements of a non-aqueous solvent and the titrant for any titration are listed, and the properties of such possible solvents are given. The potentiometric titrations of the copper - 8-hydroxyquinoline and nickel - dimethylglyoxime complexes in acetic acid, amine salts in dioxan - acetic acid and amines in $CHCl_3$ or methyl cyanide with perchloric acid in acetic acid are used to exemplify the effects. D. A. PANTONY

829. **Use of chelating agents as reagents in titrimetric analysis.** A. E. Martell and S. Chaberek (*Anal. Chem.*, 1954, **26** [11], 1692–1696).—Problems involved when complexing agents are used in titrimetric analysis are reviewed. Potentiometric and colorimetric examination of the reactions shows that unless polydentate reagents are used, formation of lower complexes, particularly near the end-point, when the ligand concn. is low, leads to gross errors. Methods of detecting the end-points *viz.*, potentiometric, colorimetric, polarographic and pH measurement, are discussed. D. A. PANTONY

830. **Photometric titrations.** R. F. Goddu and D. N. Hume (*Anal. Chem.*, 1954, **26** [11], 1740–1746).—The principles of photometric titration and its advantages and disadvantages, and the factors affecting its accuracy, are discussed; apparatus and technique are described. The end-point is determined graphically and the best choice of graph paper for the purpose is considered. A review of previous work is given. (105 references.) A. J. MEE

831. **Photometric titration of weak acids.** R. F. Goddu and D. N. Hume (*Anal. Chem.*, 1954, **26** [11], 1679–1684).—The possibilities of photometrically following the course of the titration of a weak acid (or base) with a strong base (or acid) are reviewed and compared with potentiometric methods. Curves of fraction of acid ionised against fraction titrated show that a sharp inflexion of the graph of absorption plotted against volume of titrant is obtained only when the product of the ionisation constant and molarity of the weak component is $> 10^{-12}$. The procedure is exemplified by titration of substituted phenols at various concn. (10^{-2} to 10^{-5} *M*) with 0·1 *M* NaOH. Light-absorption curves of the free acid and the phenoxide ion are examined, and a wavelength is selected at which

absorption is not excessive and, preferably, one form exhibits no absorption. The end-point is obtained graphically at the intersection of the two linear arms of the absorption - volume of titrant curves. The procedure is extended for the analysis of mixtures of two weak acids and a weak acid plus a strong acid. D. A. PANTONY

832. **Relations between voltammetry and potentiometric and amperometric titrations.** I. M. Kolthoff (*Anal. Chem.*, 1954, **26** [11], 1685–1691).—Voltammetric, potentiometric and amperometric titrations are defined and classified according to the nature of the electrodes used. Relationships between the types are discussed and the possible uses of them are reviewed. Theoretical examination of the potentiometric titration of Cu^{++} with ethylenediaminetetra-acetate at constant current shows that a large potential jump occurs just before the equivalence point, leading to large errors at low Cu^{++} concn. An expression is derived for the calculation of current changes in the neighbourhood of the end-point in an amperometric titration with two identical indicator electrodes. In general, amperometric methods have considerable advantages over potentiometric procedures.
D. A. PANTONY

833. **Use of inorganic-fibre filter-paper in gravimetric micro-analysis.** T. S. Ma, I. Kaimowitz and A. A. Benedetti-Pichler (*Mikrochim. Acta*, 1954, [6], 648–652).—The possibility of using glass fibres or fibres of vitreous silica in filter sticks, filter tubes and Gooch crucibles has been demonstrated. These substances were used in the determination of Cl' as AgCl and SO_4'' as $BaSO_4$. The use of silica-fibre filter-paper in quant. micro-analysis permits higher ignition temp. A. J. MEE

834. **Application of the ring-oven in spot colorimetry.** H. Weisz (*Mikrochim. Acta*, 1954, [6], 785–794).—In using the ring-oven method for spot-colorimetric analysis, several rings with different numbers of sample spots are prepared and compared with standard rings. Only an approx. agreement between the test and the standard rings is required to obtain usable results. The method is used for the determination of Fe with $K_4Fe(CN)_6$, Ni with dimethylglyoxime, Co with 1-nitroso-2-naphthol, Cu with H_2S solution and conversion of the CuS into its equiv. of Ag_2S. Interfering ions can normally be removed from the paper itself.
A. J. MEE

835. **Potentiometric studies in oxidation - reduction reactions. XIX. Oxidation with chloramine B.** Balwant Singh and Gurdas Singh (*Anal. Chim. Acta*, 1954, **11** [6], 569–573).—Chloramine B is a suitable reagent for the potentiometric titration of $K_4Fe(CN)_6$ (2), N_2H_4 ($\frac{1}{2}$), $FeSO_4$ (2), HI (2), As_2O_3 ($\frac{1}{2}$), quinol (1), antimonyl potassium tartrate (2) and quinhydrone (1). The bracketed figures indicate the number of molecules of the substances equiv. to 1 mol. of chloramine B. The substance for titration is mixed with dilute acid, stirred mechanically at 25° C and titrated with 0·049 *M* chloramine B with the use of a bright platinum-foil electrode and an S.C.E., the latter being connected to the soln. through an agar - KCl bridge. W. C. JOHNSON

836. **Direct quantitative spectrographic analysis of solutions.** E. V. Rouir (*Ind. Chim. Belge*, 1954, **18** [11], 1159–1170).—A critical review is given of direct spectrographic methods based either on evaporation of the soln. from an electrode followed by excitation in the arc or on direct introduction of the soln. into the arc. Factors affecting accuracy, reproducibility and convenience of operation are discussed; for these there is little to choose between procedures based on evaporation of the soln. from a carbon electrode, the Feldman hollow electrode and the rotating disc. Limits of detection by these three methods and also by flame spectrophotometry are listed for twenty-eight elements. The rotating disc is preferred as it is simple, cheap, can be adapted to any spectrographic equipment, only a min. vol. of liquid is needed and the spectra are free from background and CN-band interference. The Feldman procedure has limitations arising from its generalisation, but should become increasingly useful, although it does not require mechanical injection of a constant vol. of liquid into the arc, as with the rotating-disc method. By a two-stage procedure with the rotating-disc electrode, a complete analysis of Cu alloys can be made with an accuracy of $\pm$ 1·5 to $\pm$ 2 per cent., and of slags (after removal of SiO_2) with an accuracy of $\pm$ 4 per cent.; similarly, Ba, Sr, Ca, Mg, Sn, Si, Fe, W, Mo, Ti, Pb and Ni in oxide cathode coatings can be determined accurately even in concn. between 0·01 and 1 per cent. W. J. BAKER

837. **Non-metal analysis of micro quantities of solids by emission spectrum.** E. L. Gunn (*Anal. Chem.*, 1954, **26** [11], 1815–1819).—Production of a quantitative spectrum from non-metallic elements, particularly S and Cl, is examined in terms of ionisation potentials, time of exitation, arc-gap length, gas atmosphere, choice of internal standard, circuit inductance and design of sample-pellet holders. The sample, *e.g.*, petroleum ashes, is finely powdered and mixed with an equal wt. of internal-standard mixture [KBr (1 part) plus Ag powder (3 parts), < 200 mesh]. The final mixture is made up into a 3-layered pellet with silver powder as the centre portion. The spectrum is obtained under standard specified conditions, and the S and Cl concn. are derived from the following intensity ratios: S 3497·3 to Br 3506·5, and Cl 3622·8 to Br 3506·5. Relative accuracy on $\simeq$ 3-mg samples is given as $\simeq$ 18 per cent. on 1 to 18 per cent. S contents and 1 to 45 per cent. Cl contents. D. A. PANTONY

838. **Coulometric titrations with photometric end-point. Titration of arsenic with electrically generated iodine.** G. W. Everett and C. N. Reilley (*Anal. Chem.*, 1954, **26** [11], 1750–1753).—The possibility of using a spectrometer to detect end-points in coulometric analyses in the microgram range has been examined. A cell and associated apparatus was constructed to fit into a Beckman B spectrophotometer. The titration of arsenite with electrolytically generated iodine was investigated in this way. Before the end-point the absorbance remained at zero, but beyond the end-point the absorbance changed quickly, owing to the presence of excess of iodine. The absorbance due to the brown colour of the liberated iodine was as satisfactory as that of the blue colour with starch indicator; it was therefore unnecessary to add starch as the indicator. The end-point was obtained by plotting time against absorbance and extrapolating the linear graphs to the point of intersection. End-points could be determined easily in the microgram region. In the 50 to 100-μg range of concn., the average error was 0·5 per cent. and in the 10 to 50-μg range 2·1 per cent.
A. J. MEE

839. **Studies on the velocity of flow of solvents of some homologous series through chromatographic columns in relation to their viscosity, height of the column and density. II.** D. R. Gupta and A. K. Bhattacharya (*J. Indian Chem. Soc.*, 1954, **31** [6], 431–434).—Results of studies on aromatic hydrocarbons, aliphatic alcohols, ketones and esters indicates that $(Vh/t) \times (\eta/d)$ (where V is the volume of flow in time to a sec., h the height of column in cm, d the density and η the viscosity) gives characteristic constants for different homologous series on different adsorbents. This suggests that the flow of a solvent through surface-active columns is fundamentally connected with the structural characteristics of the solvent molecules and specific adsorption affinity of the adsorbents.
N. H. E. AHLERS

840. **Analysis of micron-sized particles.** J. P. Lodge (*Anal. Chem.*, 1954, **26** [11], 1829–1831).—"Millipore" filter material can be used to retain quantitatively particles as small as 0·2 micron in diameter. Air is drawn through the filter, which is then floated on the ion-detecting reagent or placed on a pad of blotting-paper saturated with a solution of the reagent. Nessler's solution is used for ammonium ions, ammonium ferrocyanide for Ca^{++}, *p*-nitrophenylazo-1-naphthol in 7·5 per cent. NaOH soln. for Mg^{++}, mercurous fluorosilicate for halide ions, $Pb(NO_3)_2$ for SO_4^{--} and Nitron for NO_3^-. After being washed, the filter is examined under dark-field illumination for the characteristic reaction sites. Viewing by polarised light sometimes increases contrast. A. J. MEE

841. **Extension of the gelatin method for the detection of micron-sized particles.** J. P. Lodge, jun., and H. M. Fanzoi (*Anal. Chem.*, 1954, **26** [11], 1829).—The method of Pidgeon was used to detect Ca^{++}, NO_3^- and SO_4^{--} ions in air-borne fine particles. Ca^{++}were detected by using a gelatin film sensitised with ammonium ferrocyanide; for SO_4^{--}, the gelatin was sensitised with $BaCl_2$ and for NO_3^- with Nitron.
A. J. MEE

842. **Precise measurements of particle surface area with the microscope.** F. D. Pidgeon and C. G. Dodd (*Anal. Chem.*, 1954, **26** [11], 1823–1828).—The projected-area method for determining particle surface area has been modified for direct microscopic measurement of the specific surface areas of non-porous particles, such as convex irregularly shaped crystals. Fain comparison eye-piece graticules are used to estimate directly the projected area of each particle. The method avoids the use of an empirical shape factor, thereby reducing the uncertainty of surface-area calculations based on the measurement of a diameter. The use of statistical methods for averaging size-distribution results is also eliminated. The method is rapid and requires only 2 to 6 hr. per sample; the sample must consist of particles which lie in random orientation, and procedures for ensuring this are described. Mounting techniques have been developed which permit extension of the method to subsieve-size particles. The method has been tested with crushed quartz and the surface areas obtained agree well with those arrived at from gas-adsorption and liquid-permeation methods, provided the particles are not more than 3 μ in diameter; for smaller diameters, the accuracy of the method decreases sharply. With modification, the method could be used for sub-micron particles. A. J. MEE

843. **Rapid determination of concentration of solutions with the Jelley micro-refractometer.** G. Gneisse (*Dtsch. ApothZtg.*, 1955, **95** [1], 12).—By using the Jelley micro-refractometer previously described (Kleinknecht, *Dtsch. ApothZtg.*, 1951, **91** [23], 411), the refractive index of drops of liquid may be rapidly determined within 0·001. Tables are given which relate $n_D^{20°}$ to concn. for aq. soln. of NaCl and of dextrose. A. R. ROGERS

844. **Chromatography on circular filter-paper.** O. Töppel (*Angew. Chem.*, 1954, **66** [17–18], 555–557).—The test substance is mixed with cellulose powder and packed into a tube widened at the top to form a funnel (*e.g.*, half a pipette) and with its lower end in contact with the centre of a piece of filter-paper clamped between the top and bottom flanges of a desiccator. Passage of solvent through the powder then distributes the substance more uniformly on the paper than if it were spread directly on the paper. If several substances (including a reference substance) are to be examined simultaneously, they may be placed on the paper in a ring around the central tube, which is packed with pure cellulose powder; each substance is placed on a section of the paper, which is cut into sectors to isolate the substances from one another. For development, a solution of a chloride, bromide or perchlorate of Fischer's base (1:3:3-trimethyl-2-methyleneindoline) is recommended for general work. R. C. MURRAY

845. **A simple method of applying the Beckman spectrophotometer to the measurement of paper chromatograms.** H. C. Ehrmantraut and A. Weinstock (*Biochim. Biophys. Acta*, 1954, **15** [4], 589–590).—Stained areas of chromatograms are cut into strips of the same width as the standard Beckman (Model DU) 1-cm cuvettes, but slightly shorter. These are attached to the outside of the cuvettes with adhesive Cellophane tape and cut so that when inserted into the holder the centre of the spot coincides with the centre of the light path, the area of the light-beam falling well within the coloured area of the chromatogram. With a blank paper as control, optical densities are determined in the usual way, the density being linearly proportional to the concentration. Absorption curves can be run directly on the chromatogram; this is not possible on a typical commercial densiometer.
G. W. CAMBRIDGE

846. **Observations on the utility of silver nitrate as a chromogenic agent to locate anions on paper chromatograms.** L. C. Mitchell (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 1021–1022).—Spots of AgBr, AgCl and AgI can be rendered visible by exposure to light; F can be located by subsequent heating at 100° C for 15 min. For other acids, spraying with pyrogallol to produce a contrasting background is recommended, and in some cases (*e.g.*, for fumaric, malic, succinic and tartaric acids) examination in ultra-violet light is advantageous. A. A. ELDRIDGE

847. **The separation of stereoisomeric inorganic compounds by paper chromatography.** G. Stefanović and T. Janjić (*Anal. Chim. Acta*, 1954, **11** [6], 550–553).—The cis and trans forms of dinitrobisethylenediaminocobaltic nitrate, dinitrobisethylenediaminocobaltic iodide and dichlorobisethylenediaminocobaltic chloride have been separated by paper-strip chromatography; acetone - water mixtures containing HNO_3, KI and HCl, respectively, were used as solvents. The cis form of each substance has a greater R_F value than the trans form. W. C. JOHNSON

See also Abstracts 848, 849, 860, 863, 915.

2.—INORGANIC ANALYSIS

848. **Phenylhydroxy-acids as reagents in inorganic analysis. II. Tropic acid as an inorganic analytical reagent.** A. Waksmundzki and S. Pęksa (*Ann. Univ. M. Curie-Skłodowska, AA*, 1952, **7**, 157–170).—Aq. ammonium tropate at concn. $\leq$ 2 p.p.m. gives a white ppt. with $\leq$ 50 μg of $Hg^{\cdot}$; Ag, $Hg^{\cdot\cdot}$, $UO_2^{\cdot\cdot}$, $Th^{\cdot\cdot\cdot\cdot}$, $Pb^{\cdot\cdot}$ and $Sn^{\cdot\cdot}$ interfere, but not Na, K, Mg, Zn, Cd, Ca, Sr, Ba, Al, Bi, $Cr^{\cdot\cdot\cdot}$, $Cu^{\cdot\cdot}$, $Mn^{\cdot\cdot}$, $Co^{\cdot\cdot}$, $Ni^{\cdot\cdot}$, $Tl^{\cdot}$ and $Ce^{\cdot\cdot\cdot}$. The reaction with $Hg^{\cdot}$ is quantitative and can serve for conductimetric titration; $Hg^{\cdot\cdot}$ does not interfere when $[Hg^{\cdot\cdot}]/[Hg^{\cdot}] < 0{\cdot}15$. R. Truscoe

849. **Potassium borohydride. Its use in the dry way in mineral qualitative analysis.** P. Romain, R. Merland and P. Mesnard (*Bull. Soc. Chim. France*, 1954, [10], 1298–1304).—By heating with KBH_4, oxyacids are reduced, ClO_3' and BrO_3' explosively to Cl′ and Br′, IO_3' and SO_4'' to I′ and S″, NO_3' and NO_2' to N, Phosphorus oxyacids to phosphides and PH_3, CO_3'' and C_2O_4'' to carbides, silicates to an unidentified residue and As and Sb compounds to arsenides and antimonides. Heavy-metal cations are reduced to the metal, and strongly basic cations retain their valency. The application of this reduction to each of a number of minerals is outlined and a mixture of minerals containing Fe_2O_3, SnO_2, Al_2O_3, $PbCrO_4$, $BaSO_4$ and AgCl, which would require several fusions by classical methods, is analysed quant. by a single attack with KBH_4. A general method of attack is the extraction of the powdered mineral with water and then with HCl. If the residue from the extraction is coloured, it may contain sulphides, which interfere with the detection of sulphate; these are eliminated by reaction with conc. aq. KBH_4 (on boiling for 10 min.). The final residue is then attacked by mixing with twice its weight of KBH_4, heating to spontaneous ignition and then extracting successively with water and hot *N* HCl. KBH_4 is also used to eliminate interfering ions from the ammonia group of classical qual. analysis. The ppt. in the group is dried and heated with KBH_4, the residue boiled for 5 min. with HCl to eliminate H_3BO_3 and re-tested for Fe, Al and Cr. The interfering ions are not removed quantitatively. E. J. H. Birch

850. **The selectivity of 8-hydroxy-2-methyl-5-nitrosoquinoline (2-methyl-5-nitroso-oxine) towards the group IIIB metals.** R. G. W. Hollingshead (*Chem. & Ind.*, 1954, [41], 1260).—Both 2-methyloxine and 5-nitroso-oxine are more selective than oxine itself, but are less sensitive. For 2-methyloxine, but not for 5-nitroso-oxine, non-chelation may be due to steric hindrance. The increase of acidity accompanying the nitroso group may explain the greater selectivity. In the 2-methyl-5-nitroso-oxine mol. both effects are present, and the selectivity and sensitivity of this reagent are compared with those of the other two substituted oxines for the metals Al, Y, Ga, In and Tl^{III}. 2-Methyloxine does not chelate with Al, but will chelate with Y (partial pptn. at pH 5·3), Ga, In and Tl; 5-nitroso-oxine chelates only with Tl (pptn. at pH 5·3). No chelation with any of the metals occurs with 2-methyl-5-nitroso-oxine. J. H. Waton

851. **Paper electrophoresis of inorganic substances. VII. Separation of acids in dilute hydrochloric acid as electrolyte.** M. Lederer (*Chem. & Ind.*, 1954, [48], 1481).—Movement of acids in paper electrophoresis depends on the degree of ionisation of the acid; a slow-moving acid may be either anionic or cationic in dil. HCl. With reference to radiochemical studies, separation of SO_4'' and PO_4''' ($H_2{}^{35}SO_4$ and $H_3{}^{32}PO_4$ identified on paper by a Geiger - Müller counter), SeO_3'' and TeO_3'' (detected by reduction with $SnCl_2$), and $Fe(CN)_6''''$ and $Fe(CN)_6''''$ (detected with a mixture of $Fe_2(SO_4)_3$ and $FeSO_4$) in dil. HCl was achieved. Procedures are outlined. C. A. Finch

852. **Analysis of ortho and para hydrogen mixtures by the thermal-conductivity method.** A. T. Stewart and G. L. Squires (*J. Sci. Instrum.*, 1955, **32** [1], 26–29).—Details are given of modifications to the Farkas type of thermal conductivity apparatus, which permit an accuracy of about 0·1 per cent. to be attained. G. Skirrow

853. **Colorimetric micro-determination of hydrogen peroxide.** F. Patti and P. Bonet-Maury (*Compt. Rend.*, 1954, **239** [16], 976–978).—In a method for determining very small amounts of H_2O_2, *e.g.*, in H_2O irradiated with X-rays or γ-rays, 0·5 ml of the soln. is mixed with one drop of 0·01 *N* $FeSO_4.(NH_4)_2SO_4.6H_2O$ in 3 *N* H_2SO_4, 0·25 ml of aq. 10 *M* KCNS is added and the mixture is shaken to develop the colour, the intensity of which is measured spectrophotometrically at 480 mμ. The technique is not specific for the detection of H_2O_2, as is the $Ti(SO_4)_2$ method (*Brit. Abstr. C*, 1945, 281), but it is more sensitive, *viz.*, to 0·02 μg of H_2O_2. The colour intensity of the complex remains constant at pH 3, but the stability of the aq. H_2O_2 decreases rapidly with time. W. J. Baker

854. **Assay of hydrogen peroxide in small quantities with horse-radish peroxidase as catalyst.** Y. Avi-dor, E. Cutolo and K.-G. Paul (*Acta Physiol. Scand.*, 1954, **32** [4], 314–319).—Crystallised horse-radish peroxidase (**I**) is used for determining small quantities of H_2O_2. *Procedure*—About 0·5 μg of H_2O_2 can be assayed by mixing 0·1 ml of 4-millimolar alcoholic benzidine soln., 2 ml of 0·2 *M* acetate buffer (pH 3·7), 0·2 ml of 12-micromolar **I**, an H_2O_2-containing soln. and water to 3 ml. The blue colour fades rapidly and readings are extrapolated to zero time. A semi-quant. test is more sensitive. Amberlite XE-97 (200 to 400 mesh) is suspended in 0·1 *M* acetate buffer (pH 3·7) and poured on to a sintered-glass funnel to give a layer 2 to 3 mm in thickness. Excess of buffer is removed by gentle suction, to leave a moist surface; areas of 5-mm diam. are marked with a tube. While applying gentle suction, 0·05 ml of 0·1 per cent. alcoholic benzidine soln. and 0·05 ml of 0·4 to 0·6 m*M* **I** in water are put on the spots and the H_2O_2-containing soln. ist hen applied; 0·003 μg of H_2O_2 in 2 ml of soln. gave a distinct blue spot, whereas half this quantity did not. The concn. of H_2O_2 is obtained by diluting the soln. of unknown strength with H_2O until 2 ml of it give a spot of $\simeq$ equal intensity to 2 ml of 5×10^{-8} *M* H_2O_2. H. F. W. Kirkpatrick

855. **Modified high-frequency apparatus for the determination of moisture in solids. Determination of moisture in sodium chloride and ammonium nitrate.** F. W. Jensen, M. J. Kelly and M. B. Burton, jun. (*Anal. Chem.*, 1954, **26** [11], 1716–1719).—A high-frequency method of determining the moisture in NaCl and NH_4NO_3 is based on the change in the properties of a mixed solvent system due to the equilibrium extraction of the moisture and salt from the solid. An ideal solvent is one which dissolves water well and enough of the salt

to give an appreciable, but not excessive, conductivity. A mixture of 1:4-dioxan and methanol gave the best results for the salts used. The composition of the optimum mixture is different for the two salts. The apparatus is described. The method has a precision of 0·02 per cent. of moisture in the solid sample for the salts used. A. J. MEE

856. **Simple and rapid method for the identification of cations of the first analytical group.** D. Markovics (*Gyógyszerész*, 1954, **9** [1], 16–17; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 31,081).—Potassium iodide is used for the identification of cations of the first analytical group. The iodides of Ag, Hg, Pb, Cu and Bi differ not only in their appearance but also in their behaviour with excess of potassium iodide and with other reagents.
E. HAYES

857. **Separation of alkali chloride from ammonium chloride using anion-exchange resin.** T. Murase, Y. Sasaki and H. Kakihana (*J. Chem. Soc. Japan*, 1954, **75** [2], 227).—To eliminate the possible loss of alkali chloride (especially of CsCl and RbCl) on the sublimation of ammonium chloride, the following method is recommended. The 20 to 50-mesh resin Amberlite IRA-410 should first be treated successively, several times, with *N* NaCl and *N* NaOH. Immediately before use, the column (int. diam., 1 cm; length, 22 cm) is washed thoroughly by CO_2-free water (3 ml per min.). The aq. soln. of mixed chlorides is passed through the column (1·5 ml per min.) and Cl' is replaced by OH'. By evaporating the filtrate, NH_3 is easily expelled; alkali metals are determined in the residue. To remove the last trace of ammonium carbonate from the residue, it is heated on a water-bath. This method is satisfactorily applied to the analysis of alkali metals in natural substances. K. SAITO

858. **Analytical methods used in studies on continuous separation of ions by countercurrent ion exchange. [The determination of lithium and potassium.]** S. B. Radding, R. C. Phillips and N. K. Hiester (*Anal. Chim. Acta*, 1954, **11** [6], 538–549).—In the determination of Li and K by the flame photometer, the optimum slit-width is investigated. The presence of HCl has an effect on results and the two metals exhibit some mutual interference. Dry or wet-ashing of a resin followed by flame-photometric measurements does not yield satisfactory results for the determination of Li and K. Elution with *N* or 2 *N* HCl is preferred. Several methods for assessing the exchange capacity of a resin are described. The preferred method consists in treating the resin with aq. $CaCl_2$ (5 per cent.) and titrating the resultant acidity of the solution. W. C. JOHNSON

859. **Spectroscopic assay of lithium isotopes. I. Measurements at high intensity. II. Measurements on small samples at low intensity.** J. K. Brody, M. Fred and F. S. Tomkins (*Spectrochim. Acta*, 1954, **6** [5–6], 383–412).—**I.** A method for determining the natural ^7Li to ^6Li abundance ratio by measurements of the resonance line of Li I at relatively high temp. is described. The 6707 A lines are resolved either by the 30-ft. Paschen-Runge grating spectrograph or by the Fabry-Perot interferometer with the grating spectrograph as a monochromator. The self-absorption correction is calculated from theory and observations on the ^7Li doublet intensity-ratio. A liquid-air-cooled hollow cathode source is used to reduce the Doppler broadening. The problems involved in making accurate intensity measurements with both high-resolution instruments are discussed and the necessary instrumental corrections are indicated. The spectroscopic analysis is much more difficult than for uranium isotopes, mainly because the line-width approaches the magnitude of the isotope shift. The mean value of ^7Li to ^6Li for normal Li in laboratory reagents was $13{\cdot}5 \pm 0{\cdot}2$, the values being approx. constant with intensity, except at high intensities.

II. The above grating procedure is modified so that the measurements can be made on 2-μg samples, and the self-absorption and line-broadening reduced. To ensure an adequate line-to-background ratio, the Li sample is diluted with KCl and placed in a hollow aluminium cathode having a water-proofed graphite insert. That the 6707 A lines can be resolved without self-absorption is shown by the observed doublet intensity-ratio of $2{\cdot}04 \pm 0{\cdot}02$ (theory gives 2·00). Values of the intensity ratio for mixtures of separated isotopes are identical with the abundance ratio in the range of normal Li.
W. J. BAKER

860. **Determination of potassium by means of sodiumtetraphenylboron.** A. F. Ievin'sh and É. Yu. Gudrinietse (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 270–274).—An improved method of synthesis of sodiumtetraphenylboron and the use of this reagent in a new volumetric method for determining K are described. After pptn. of the potassium compound at pH 3 to 6, the excess of reagent is pptd. by the addition of ammonium chloride and the excess of ammonium ion is found by titration. G. S. SMITH

861. **Paper chromatography of inorganic ions. IX. A note on the preparation of carrier-free ^{131}Cs.** M. Lederer (*Anal. Chim. Acta*, 1954, **11** [6], 528–529).—To separate ^{131}Cs from irradiated $BaCO_3$, the material is dissolved in aq. HCl, the $BaCl_2$ is crystallised and the ^{131}Cs is concentrated in a small quantity of mother liquor. The liquor is then subjected to paper chromatography; phenol saturated with 2 *N* HCl is used as solvent. W. C. JOHNSON

862. **Study of the possibility of preventing the co-precipitation of copper with iron hydroxide.** I. L. Teodorovich and B. V. Rakhimova (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 293–298).—The addition of glycine or ethylenediamine almost eliminates the co-pptn. of Cu with $Fe(OH)_3$, when pptn. with ammonia is carried out in the cold.
G. S. SMITH

863. **Thiobenzamide as an analytical reagent [for copper].** E. Gagliardi and W. Haas (*Mikrochim. Acta*, 1954, [6], 593–598).—Thiobenzamide gives a yellow complex with Cu salts; the colour is given strongly in neutral or HCl solutions. In 15 *N* H_2SO_4, a yellow ring can be observed, but it is weak in 20 *N* H_2SO_4. In HNO_3, the intensity of the colour is stronger in 0·1 *N* than in *N* solution. The strongest colour is obtained in *N* HCl. The yellow ring is not easily seen in 20 per cent. acetic acid and on heating a brown to black ppt. is formed. The reaction can be used as a spot test for Cu; 0·5 μg of Cu can be detected. The yellow complex obeys the Beer-Lambert law and the reaction can be used for the colorimetric determination of Cu. Alkali metal and ammonium nitrates, chlorides or acetates, and tartaric and phosphoric acids do not affect the extinction, even in 1000-fold excess; metallic salts that do not give a colour in HCl

solution also have no effect. Coloured salt solutions must be decolorised, or the substance must be removed. A. J. MEE

864. Glutamic acid as an addition agent in the electro-deposition of copper. S. Adamek and C. A. Winkler (*Canad. J. Chem.*, 1954, **32** [10], 931–940).—The polarisation - time curves for copper obtained in the presence of glutamic acid generally show two distinct polarisation levels above the standard surface value; the first corresponds to an induction period. Polarisation at both levels increases with increase of glutamic acid concn. and decreases with increase of H_2SO_4 concn. in the electrolyte. In general, the induction period increases and eventually becomes non-reproducible with increased H_2SO_4 and $CuSO_4$ concn., decreased glutamic acid concn., decreased current density, increased temp. and addition of chloride. Addition of sufficient chloride prolongs the induction period indefinitely at a polarisation level corresponding to the presence of chloride alone. After the induction period, the concn. polarisation increases with glutamic acid concn. and is considerably higher than the value obtained during the induction period. The addition of chloride decreases the concn. polarisation. D. BAILEY

865. Separation and gravimetric determination of copper in the presence of iron or aluminium or both metals. Gh. Spacu and Despina Pirtea (*Comun. Acad. R.P. Române*, 1953, **3** [1–2], 77–82; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 31,099).—Copper is pptd. as $CuPy_2(CNS)_2$ (Spacu and Dick, *Z. anal. Chem.*, 1927, **71**, 185) from a soln. containing tartrate, which prevents the pptn. of Fe and Al. Fe and Al are pptd. from the filtrate by an ethanolic soln. of 8-hydroxyquinoline. E. HAYES

866. Spectrochemical determination of copper in crank-case drainings. C. R. Hodgkins and J. Hansen (*Anal. Chem.*, 1954, **26** [11], 1759–1762).—One gram of well-shaken oil is ignited in the presence of a buffer - internal-standard mixture [100 g of "Calcium sulphonate" (mol. wt. 900, 30 per cent. soap, in oil), 8 g of "Lead naphthenate" (24 per cent. lead, in oil) and 0·34 g of Co, as cobalt naphthenate, in 500 ml of xylene] (3 g); the ignition is completed at 1000° to 1200° F. The ash (1 pt.) is ground with Li_2CO_3 - graphite mixture (1 + 1) (9 pt.) and then packed into specially shaped sample electrodes. The determination is completed by a standard spectrographic procedure over the range 3000 to 3300 A at an exposure time of 75 sec. Synthetic standards are treated similarly, and the Cu concn. is calculated from the following intensity ratios: Cu 3247·5 to Co 3044·0, and Cu 3274·0 to Co 3044·0. Results are compared with those from standard chemical analysis. D. A. PANTONY

867. Paper electro-chromatography. II. Behaviour of silver salts and the separation of silver, mercury and lead salts. S. Harasawa and T. Sakamoto (*J. Chem. Soc. Japan*, 1954, **75** [2], 229).—Paper electro-chromatographic behaviour of Ag^+ in 3 *N* aq. NH_3 soln., 30 per cent. formic acid, 0·5 *M* citric acid, 0·5 *M* lactic acid, 0·5 *M* acetic acid and 0·5 *M* tartaric acid solution was studied under an electric current of 500 V and $\simeq$ 3 mA. Ag^+ in aq. NH_3 soln. moves in the form of $Ag(NH_3)_2^+$. In organic acid soln., Ag^+ shows double spots on paper. When different electric potentials are applied (100 to 500 V), the migration distance is approx. proportional to the voltage. In organic acid soln., Ag^+, Hg^+ and Pb^{++} can be detected separately under 500 V and approx. 4 mA in 30 min. K. SAITO

868. Separation of small amounts of silver from lead by extraction with dithio-β-isoindigo. E. Gagliardi, M. Theis and W. Klementschitz (*Mikrochim. Acta*, 1954, [6], 653–656).—Small amounts of Ag can be separated quantitatively from a Ag - Pb solution by the addition of a solution of dithio-β-isoindigo in *n*-butanol. The ppt. formed with Ag dissolves in butanol to give a dark red solution. The butanol layer is separated from the aq. phase, which contains all the Pb, the Ag complex is decomposed with HNO_3 and the Ag is again extracted with H_2O from the butanol solution. The Ag is then determined by titration with NH_4CNS and the Pb in the aq. extract is determined with complexone III. About 1 hr. is required for the determination. Results are generally satisfactory. A. J. MEE

869. Effect of varying the temperature and the relative amount of lead used in determining gold by cupellation. A. Popović (*Bull. Soc. Chim., Belgrade*, 1954, **19** [5], 309–313).—Large excesses (> 50 per cent.) of Pb cause low results in the cupellation of alloys containing < 50 per cent. of Au; the finer the gold, the less is the difference on taking an excess (up to 100 per cent.) of Pb. Cupellation should begin at 750° C, the temp. gradually being raised to 1000° C. R. TRUSCOE

870. A method for the separation and identification of the common group-2 elements. P. Heath (*Analyst*, 1954, **79**, 781–783).—In the suggested method, the sulphides are pptd. and washed in the usual way. The ppt. is boiled with conc. HCl and the cooled suspension is saturated with H_2S. The soln. may contain Cd, Bi, Sb, Sn^{II}, Sn^{IV} and small amounts of Pb, and the ppt., Cu, As and Hg. The ppt. is dissolved in aqua regia and S is removed. The soln. is then diluted and made alkaline with aq. NH_3. Arsenic is pptd. as crystalline magnesium ammonium arsenate by the addition of conc. $MgCl_2$ soln. and the ppt. is collected. Cu and Hg are detected in the filtrate by the usual confirmatory tests. The acid filtrate from the sulphide separation is evaporated to low bulk, a few drops of H_2SO_4 are added and after a few min. the liquid is diluted slightly and the pptd. $PbSO_4$ removed. A small portion of the filtrate is tested for Sn which, if present, is oxidised with Br water and the excess of Br is removed by boiling. The soln. is then treated with aq. NH_3 and saturated with H_2S; Bi and Cd are pptd. and removed. Sb and Sn remain in soln. as thio salts. The ppt. is dissolved in HCl, H_2S is removed and an excess of aq. NH_3 is added. Bi is pptd. and Cd remains in solution. Appropriate confirmatory tests are described for application to each separated group of metals. A. O. JONES

871. Absorptiometric determination of magnesium in titanium and its alloys. H. J. G. Challis and D. F. Wood (*Analyst*, 1954, **79**, 762–770).—Methods are described for the determination of Mg in Ti and its alloys. For Ti metal or sponge, the Ti is separated by extraction of its cupferron complex with $CHCl_3$. After the removal of excess of cupferron, the aq. soln. is treated with Titan yellow in the presence of glycerol and hydroxylamine hydrochloride, with starch as protective colloid, and the optical density is measured in a Spekker absorptiometer with an Ilford No. 605 yellow filter against the untreated aq. soln. as blank. The Mg content

is then ascertained from a previously constructed calibration graph. With Ti alloys, the Ti and alloying elements are removed by separations involving the use of cupferron, ZnO - $KMnO_4$ and NaOH - KCN. The Mg is then determined absorptiometrically by the Solochrome cyanine method (Bacon, *Brit. Abstr. C*, 1952, 134). A. O. JONES

872. **Photometric determination of magnesium in electronic nickel.** C. L. Luke and M. E. Campbell (*Anal. Chem.*, 1954, **26** [11], 1778–1780).—A 0·01 to 0·1 g sample of Ni containing 0·1 to 0·001 per cent. of Mg is dissolved in warm dil. HNO_3 (1 + 1) (2 ml). To the soln. is added aq. $Fe(NO_3)_3$ (0·1 per cent. as Fe) (1 ml) and $\simeq$ 40 ml of water. Aq. NH_3 is added to 1 ml in excess of neutrality (Congo red), and the suspension is digested at 65° to 70° C. The ppt. is separated by filtration, but not washed, and to the filtrate is added 5 per cent. oxalic acid (2 ml); H_2S is passed in for 5 min. and the solution is filtered as before. The filtrate is boiled (20 min.), and cooled, and to it is added 4 per cent. sodium diethyldithiocarbamate. After being allowed to stand (10 min.), the suspension is filtered and the ppt. is washed with the minimum amount of water. The vol. of filtrate and washing is adjusted to 40 $\pm$ 2 ml with water, and the Mg^{++} is extracted by addition of 2-butoxyethanol (5 ml), aq. NH_3 (10 ml) and 8-hydroxyquinoline (3 per cent. in $CHCl_3$) (20 ml) and shaking for 1 min. in a separating funnel. Eighteen ml of the $CHCl_3$ are filtered and examined photometrically at 400 mμ with reference to a reagent blank (1-cm cells). Mg concn. is calculated from a calibration curve derived from standards. Examples of analytical figures for the method are given. D. A. PANTONY

873. **A rapid flame-photometric method for the determination of calcium in coal ash and coke ash.** L. J. Edgcombe and D. R. Hewett (*Analyst*, 1954, **79**, 755–758).—A method for the rapid determination of Ca in coal ash and coke ash is described. Si is first removed by treatment of the ash (0·1 g) with HF and H_2SO_4. The residue is dissolved by the addition of boiling water and the vol. of the cooled soln. is adjusted to 100 ml. An aliquot portion (20 ml) is made just alkaline to methyl red with aq. NH_3 and centrifuged, the clear supernatant liquid is then decanted into a 50-ml flask. The residue is treated with 5 ml of dil. H_2SO_4 and $\simeq$ 10 ml of water and the pptn. and centrifuging are repeated. The vol. of the combined extracts is adjusted to 50 ml and the Ca is determined with a flame photometer by direct comparison with a standard soln. containing 50 p.p.m. of CaO. The extent to which the more common elements present in the final soln. and the acid radicles introduced interfere has been determined and methods are described for eliminating the interfering elements or for making allowance for their presence. A. O. JONES

874. **Spectroscopic determination of strontium and lithium in natural waters.** T. F. Borovik-Romanova, V. V. Korolev and Yu. I. Kutsenko (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 265–269).—The Sventitskiĭ activated a.c. arc and a quartz fulgurator (described and illustrated) are used for the determination of Sr and Li in natural waters. G. S. SMITH

875. **A compleximetric method for the determination of zinc in aluminium alloys.** J. C. Sergeant (*Metallurgia*, 1954, **50**, 252–254).—A simple and speedy method for the routine analysis of Zn in Al alloys is described. *Procedure*—To the sample are added nitric and hydrochloric acids and, after boiling and diluting, tartaric acid. After neutralisation with ammonia solution, the solution is re-acidified with dilute nitric acid; sodium carbonate - sodium sulphide solution is then added to neutralise the nitric acid. After again boiling, the combined precipitates of Zn, Cu, Ni, etc., are filtered and washed with dilute carbonate - sulphide solution. The filtrate is transferred to the original beaker, ammonium chloride and bromine water are added, the solution is boiled and made just ammoniacal, to precipitate Fe, Al, etc. These precipitates are filtered off and washed with ammonium chloride solution and the filtrate is transferred to a titration beaker. Further ammonia is added, any remaining Cu and Ni are complexed with potassium cyanide solution and the $Zn(CN)_2$ is decomposed by formaldehyde. The sample is then titrated with EDTA with Eriochrome black as indicator. G. C. JONES

876. **Use of 8-hydroxyquinoline for determination of zinc in solutions containing copper.** S. Z. Haider and M. H. Khundkar (*Analyst*, 1954, **79**, 783–784).—A method is described for the separation of Zn from Cu by 8-hydroxyquinoline. The soln. is treated with an excess of KI and the Cu is determined by titration with standard $Na_2S_2O_3$ soln. Thiourea is then added until the Cu_2I_2 ppt. dissolves, the soln. is acidified with acetic acid and $\simeq$ 5 g of sodium acetate are added. The soln. is heated to 60° C and alcoholic 8-hydroxyquinoline soln. is added to slight excess, heating being continued until the ppt. becomes granular. The ppt. collected on a tared sintered-glass crucible is washed with warm water, dried at 130° to 140° C and weighed. Alternatively, the ppt. is dissolved in 2 *N* HCl and diluted to a known vol. and aliquots are titrated with standard $KBrO_3$ soln. with methyl red as indicator, or excess of $KBrO_3$ may be added and the excess determined. A. O. JONES

877. **Quantitative determination of mercury with thiobenzamide.** E. Gagliardi and W. Haas (*Mikrochim. Acta*, 1954, [6], 599–603).—The coloration produced with Hg^{II} salts in HNO_3 and thiobenzamide depends on the concn. of the Hg^{II} ion. In solutions that contain < 14·7 mg of Hg in 10 ml, the addition of a 1 per cent. solution of thiobenzamide in 60 per cent. ethanol and boiling gives a black ppt. or, with dilute solutions, a black colloidal solution. If the solution contains > 14·7 mg of Hg in 10 ml, the ppt. is yellow. For a concn. of > 17 mg of Hg in 10 ml, a white to creamish-white colloidal solution is formed, and for > 20 mg of Hg in 10 ml, a white flocculent ppt. The colour changes are sharp and are reproducible; the reaction is therefore suitable for the rapid determination of Hg. NH_4Cl, NaCl, KCl and KBr interfere. If the Hg is present as the KI complex, no reaction takes place with thiobenzamide. NH_4NO_3 and $NaNO_3$ do not interfere and nitrates of the following elements do not interfere: Al, Fe, Cr, Ni, Co, Mn, Zn, Cd, Mg, Ca, Sr and Ba. If the concn. of HNO_3 is increased, the colour changes are less sharp. The analysis can be carried out rapidly, 10 to 15 min. being sufficient. The average error is $\pm$ 0·26 per cent. A. J. MEE

878. **Determination of mercury with 2-mercaptobenzothiazole.** I. Ubaldini and F. Capizzi Maitan (*Ann. Chim., Roma*, 1954, **44** [7–8], 530–533).—The mercury salt is dissolved in an excess of KI soln. and a solution of 2-mercaptobenzothiazole in aq. KOH is added; after filtration and drying at

105° C, the ppt. is weighed. The compound $[Hg(C_6H_4CNS_2)_2]$ contains 37·63 per cent. of Hg. The min. concn. detectable in a qual. test is 0·5 μg of Hg per ml. Good agreement is found between weights of Hg taken (0·005 to 0·103 g) and those found. R. C. MURRAY

879. **The removal of dissolved carbon dioxide in the volumetric determination of boron.** H. Jackson and R. E. Bailey (*Analyst*, 1954, **79**, 785).—In the potentiometric titration of H_3BO_3 with NaOH soln. between pH 5·7 and 8·6 or in the visual titration with suitable indicators in the presence of mannitol, any dissolved CO_2 must first be removed by boiling. This process may be tedious and may involve loss of H_3BO_3 by volatilisation. The CO_2 can be effectively removed by adjusting the pH to $\simeq$ 3 and bubbling a vigorous stream (2·5 to 3 litres per min.) of filtered compressed air through the soln. before the titration. A. O. JONES

880. **Analysis of electroplating solutions. II. Estimation of boric acid in the presence of nickel and ammonium salts.** M. R. Verma and K. C. Agrawal (*Electroplating*, 1954, **7** [11], 403–404).—A method is described for the determination of boric acid in the presence of Ni and ammonium salts by treating the sample with formaldehyde. An excess of formaldehyde and potassium oxalate is added to the solution, which is then titrated to a definite pH value so that any free acid already present or that obtained from the ammonium salts is neutralised. Mannitol is then added and the titration is continued, the amount of alkali consumed in the second stage being noted. When Ni salts and boric acid are present in the molecular ratios met with in normal practice, the values are accurate to $\pm$ 2 per cent. The method is simple, speedy and suitable for routine determinations. G. C. JONES

881. **Method for determining the carbide content of synthetic corundum.** Miklós Ferenczy (*Kohászati Lapok* (*Aluminium*), 1953, **8** [3], 49–52; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 27,560).—A weighed sample (0·5 g) of synthetic powdered corundum is mixed with PbO_2 and $K_2Cr_2O_7$ and heated in a stream of oxygen at 900° C. The CO_2 formed by the oxidation of Al_4C_3 is entrained in a 20 per cent. soln. of KOH and measured by weighing; in an alternative method, the CO_2 is absorbed by 0·1 *N* $Ba(OH)_2$ and the pptd. $BaCO_3$ is filtered off and titrated with 0·1 *N* HCl. Results obtained by both methods agree within $\simeq$ 0·3 per cent. E. HAYES

882. **Observations on the technique of fractional crystallisation of the binary magnesium nitrates of the rare-earth elements.** G. Virt-s (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 299–303).—The following three methods of crystallisation of the binary magnesium nitrates of the Ce group are studied: spontaneous crystallisation with slow cooling of the hot solution in air without stirring; seeding of the hot solution and slow cooling in air without stirring; and seeding of the hot solution and cooling with vigorous stirring. The third method is the most effective, particularly if cooling is slow. Radioactive Eu is used to indicate the effectiveness of the separation. G. S. SMITH

883. **Spectrophotometric determination of some rare earths and yttrium with alizarin red S.** R. W. Rinehart (*Anal. Chem.*, 1954, **26** [11], 1820–1822).—A chloride or nitrate soln. of a rare-earth metal (10 to 100 μg per ml) or yttrium (8 to 80 μg per ml) is prepared either by the evaporation to dryness of an acid soln. to expel excess of acid, or an oxalate compound (*e.g.*, mounted on filter-paper) is ashed and ignited at 900° C and the residue dissolved in dil. HCl; the soln. are made up to vol. A sample of 3 to 5 ml of the soln. is made just alkaline with 0·2 *N* NaOH (phenol red), and then just acid with 0·03 *N* HCl; to this are added buffer soln. (2 *N* aq. ammonium acetate - 2 *N* acetic acid) (0·5 ml) and 0·1 per cent. aq. alizarin red S (2 ml), and the soln. is made up to 10 ml. After 5 min., the absorption is measured against a reagent blank at 550 mμ (1-cm cell), and the rare-earth concn. is calculated from calibration curves derived from standards. The method is non-specific. Figures obtained in the analysis of Nd are given. D. A. PANTONY

884. **Micro-titrations with ethylenediaminetetra-acetic acid. XII. Determination of gallium.** H. Flaschka and H. Abdine (*Mikrochim. Acta*, 1954, [6], 657–667).—The theory of the complexiметric titration of Ga is discussed. Ga can be determined by the addition of excess of complexone III to the solution and back-titrating with Zn (indicator: Eriochrome black T or naphthidine), Pb (indicator: Eriochrome black T) or Cu [indicator: murexide or 1-(2-pyridylazo)-2-naphthol]. Ga may be determined with the use of Pb solutions for back-titrating the excess of complexone III in the presence of Zn, Cu, Co, Ni or Fe, if these elements are masked with KCN. Ga can also be determined by back-titrating with Zn or Cu at pH $<$ 6, when large amounts of alkaline earths are present. A. J. MEE

885. **Potentiometric method for the macro- and micro-determination of thallium by oxidation with potassium permanganate in alkaline solution.** I. M. Issa and R. M. Issa (*Analyst*, 1954, **79**, 771–775).—A method is described for the potentiometric determination of Tl depending upon the oxidation of Tl^{I} to Tl^{III} by $KMnO_4$ in alkaline soln. ($\simeq$ 0·1 *N* NaOH). The titration device has been described previously (Issa *et al.*, *Brit. Abstr. C*, 1953, 463). Tl^{III}, if present, is first reduced with SO_2 in acid soln., the unused SO_2 being expelled by passing CO_2 through the heated soln. The method can be used for determination of 5 μg of Tl. A. O. JONES

886. **Precision of mass-spectrometer analyses of carburetted water gas.** W. Volk (*Anal. Chem.*, 1954, **26** [11], 1771–1774).—The results obtained from co-operative mass-spectrometer analyses by twenty seven laboratories of a standard sample of carburetted water gas was reviewed to determine the precisions within the laboratory and between the laboratories and the sources of imprecision in the mass-spectrometer operation. The precision attained within each laboratory was from $\pm$ 0·2 to $\pm$ 0·6 per cent. and an average check between laboratories of $\pm$ 0·3 to $\pm$ 5·9 per cent., depending on the gas component analysed. The main variations were caused by operator and spectrogram error, the results available not permitting clear differentiation between the two causes. Proposals for the improvement of precision are given. G. P. COOK

887. **Analysis of flue gases with a modified Haldane apparatus.** R. M. Simpson (*Chem. & Ind.*, 1954, [46], 1415–1416).—The apparatus is used to analyse flue gases, which contain $\simeq$ 8 per cent. of CO_2, 7 per cent. of CO, 10 per cent. of H and 0·1 per cent. of O, the remainder being N, from a plant producing lampblack from creosote. Because of the high CO_2 content, a second arm filled with 1 per cent. H_2SO_4 is included in the standard pipette and KOH is only used for CO_2 removal.

Combustion is carried out with O in preference to air. High concn. of SO_2 would interfere with this method. A. M. SPRATT

888. **Colorimetric determination of carbonyl sulphide in synthesis gas.** L. A. Pursglove and H. W. Wainwright (*Anal. Chem.*, 1954, **26** [11], 1835–1839).—The method is based on the hydrolysis of COS with dilute base and the determination of S'' by the methylene-blue procedure. As CO_2 and H_2S interfere with the procedure, the gas is passed through 40 per cent. KOH soln. and over heated Fe_2O_3 before sampling. The COS in the sample is absorbed on shaking with 0·1 *N* KOH and the methylene-blue procedure is then applied. Carbon disulphide, ethanethiol and thiophen do not interfere. Reproducibility is good, a range of $<.0{\cdot}12$ g being obtained at the 1·5-g level of COS per 100 cu. ft. of gas. G. P. COOK

889. **Silica gel for use as desiccant for packages.** British Standards Institution (B.S. 2540: 1954, 10 pp.).—The pH, particle size, friability and absorptive capacity and content of ammonia, ammonium compounds, soluble chloride, soluble sulphate and water are prescribed for packages; methods of determining these properties are also given. H. P. PAGET

890. **Spectrochemical analysis of silicate by a large glass spectrograph. I. Construction of the spectrograph and the method of analysis.** C. Iida and K. Yamazaki (*J. Chem. Soc. Japan*, 1954, **75** [2], 189).—A Mannkopf-type spectrograph with two glass prisms was constructed to have a resolving power of 3·4 A per mm at 3800 A and 10 A per mm at 4800A. Rough quant. analyses of minor constituents in silicates were carried out by the cathode-layer method. The sample ($\simeq$ 9 mg) is put into a hole of a carbon electrode and burnt in a d.c. arc (180 V; 180-mH 48-μF stabiliser). Four spectrograms are taken successively at currents of 5, 7, 9 and 9 amp. (exposure, 45 sec. each). The base substance for standard samples consists of 77 per cent. of SiO_2, 20 per cent. of Al_2O_3 and 3 per cent. of Fe_2O_3. The contents of Ba, Co, Cr, La, Mn, Ni, Pb, Sr, V and Zr were estimated by comparing the approx. intensity of the lines with the aid of a step-sector. K. SAITO

891. **Use of oscillographic polarography for quantitative determination of titanium.** Ya P. Gokhshtein, S. I. Sinyakova and V. D. Yukhtanova (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 255–264).—The reduction of Ti^{IV} in acid medium on the dropping-mercury electrode is studied by means of the oscillographic polarograph described previously (*Anal. Abstr.*, 1954, **1**, 1746). In *N* H_2SO_4, the tartrate and citrate complexes of Ti are unstable, but the oxalate complex is stable. In acid medium saturated with Na oxalate, Ti is reduced in the anionic form. The relationship between max. current and concn. is linear. Cr and Ni have no effect on the height of the oscillographic wave, nor have Fe up to 100 times or V up to 20 times the Ti concn. Mo interferes seriously, but if its content is known it can be allowed for in constructing calibration curves. The method is applied to the determination of Ti in steels containing V, without preliminary separation of Fe and V. G. S. SMITH

892. **Standardisation of titanous solutions against potassium dichromate.** R. H. Pierson and E. St. C. Gantz (*Anal. Chem.*, 1954, **26** [11], 1809–1811).—The advantages of using $K_2Cr_2O_7$ as primary standard for the determination of $Ti^{\cdots}$ in soln. are discussed. Details of procedures with 0·2 *N* $K_2Cr_2O_7$ under CO_2 with sodium diphenylbenzidine-sulphonate, diphenylaminesulphonate or phenylanthranilic acid as indicator are given. *Procedure for the determination of the Fe content of the* Ti^{III} *soln.*—The Ti^{III} soln. (40 ml) is treated with dil. H_2SO_4 (1 + 10) (80 ml), a moderate excess of 0·1 *N* $KMnO_4$ and then 20 per cent. aq. NH_4CNS to destroy the excess of $KMnO_4$, when a further 3 ml are added; the resulting Fe^{III} is titrated with the previously standardised Ti^{III} soln. The Fe content is calculated from a given equation. It is suggested that Ti^{III} soln. are best prepared from the hydride, which can be obtained almost free from iron. D. A. PANTONY

893. **Amperometric titration of zirconium. Application to fluoride solution.** E. C. Olson and P. J. Elving (*Anal. Chem.*, 1954, **26** [11], 1747–1750).—Five to ten ml of a solution containing 0·02 to 0·1 millimole of Zr obtained by dissolving Zr metal in H_2SO_4 - HF mixtures is treated with 1 ml of 1 per cent. aq. gelatin and diluted to 50 ml with dil. (1 + 10) H_2SO_4. Under nitrogen, freshly prepared standard 0·05 to 0·1 *M* aq. cupferron is added in small portions and the titration is followed amperometrically at a potential of $-$ 0·84 V with respect to the S.C.E. Intersection of the two linear arms of the current - volume of titrant graph gives the end-point. Excessive F' (> 35-fold excess) is removed by the addition of 0·4 *M* $Al^{\cdots}$ in dil. (1 + 10) H_2SO_4 (50 ml). Precision is given as $\pm$ 0·37 per cent. Results compare favourably with those from a standard cupferron-pptn. gravimetric procedure. $Fe^{\cdots}$, $Ti^{\cdots\cdot}$, V^{v}, $Sn^{\cdots\cdot}$ and $Hf^{\cdots\cdot}$ interfere, but PO_4''' does not, provided that 5 to 10 min. elapse between the addition of the cupferron soln. and the reading of the current. D. A. PANTONY

894. **Spectrochemical determination of thorium in monazite by the powder d.c. arc technique.** C. V. Dutra and K. J. Murata (*Spectrochim. Acta*, 1954, **6** [5–6], 373–382).—In the method described, Zr is used as internal standard; the analytical curve for Th^{II} 2870·413 A - Zr^{II} 2844·579 A is established by means of synthetic standards containing graduated amounts of ThO_2 and 0·500 per cent. of ZrO_2 in pegmatite (quartz 60 pt., microcline 40pt., Fe_2O_3 1 pt.). The monazite samples are diluted 14-fold with pegmatite-base containing 0·538 per cent. of ZrO_2, so that the ZrO_2 content of the mixture is also 0·500 per cent. The standards and the diluted monazites are then mixed with one-half of their wt. of powdered graphite. Approx. 25 mg of the prepared samples are arced to completion at 15·5 to 17·5 amp. The results are accurate for ThO_2 contents of 3 to 20 per cent. in the original monazite. The coeff. of variation is $\simeq$ 4 per cent. for a ThO_2 content of $\simeq$ 7 per cent.; this represents improved precision in comparison with the total-energy method and carefully weighed samples. Tests with synthetic mixtures and chemically analysed monazites show a max. error of $\pm$ 10 per cent. of the ThO_2 content. The method can be used to determine the zonal distribution of ThO_2 in monazite crystals. Neither Nb nor Pt is satisfactory as internal standard. W. J. BAKER

895. **Coulometric and polarographic determinations of trace amounts of lead.** T. L. Marple and L. B. Rogers (*Anal. Chim. Acta*, 1954, **11** [6], 574–585).—A stationary mercury-plated platinum electrode in a rapidly stirred soln. is applied to the

polarographic and coulometric determination of Pb in micromolar concn. Stirring produces a six-fold increase in the polarographic wave height and a two-fold increase as compared with the dropping-mercury electrode. The coulometric method depends upon the deposition and re-solution of Pb, according to the principle previously used for the determination of Ag (*Brit. Abstr. C*, 1952, 2186); concn. of Pb down to 10^{-8} *M* can be dealt with. W. C. JOHNSON

896. **A possible source of error in the determination of trace metals, particularly lead.** J. G. Maltby (*Analyst*, 1954, **79**, 786–787).—The enamel for filling the graduation marks on one make of graduated pipettes now contains Pb, which is extracted by ammonium citrate soln. in Pb determinations in sufficient amount to contaminate the lead-free soln. As the graduated pipettes used frequently have the lower graduations immersed in the liquid being measured, contamination with Pb may occur unless the enamel is removed from the graduations. A. O. JONES

897. **Paper chromatography of inorganic ions. VIII. Separation of cations from phosphate ions.** M. Lederer (*Anal. Chim. Acta*, 1954, **11** [6], 524–527).—Pyro- and meta-phosphate ions, as well as the ions of various condensed phosphates, yield comet-like chromatograms on paper, when butanol saturated with *N* HCl is used as solvent; this effect is attributed to partial conversion to orthophosphate during the process. Orthophosphate, arsenate and arsenite yield normal chromatograms. The same procedure is used for the separation of $Fe^{\cdots}$, $Cu^{\cdot\cdot}$ and $UO_2^{\cdot\cdot}$ from $PO_4^{\prime\prime\prime}$ in micro and macro amounts. W. C. JOHNSON

898. **Colorimetric determination of phosphorus and arsenic compounds. I. Phosphorus compounds.** G. Di Bacco (*Boll. Chim. Farm.*, 1954, **93** [2], 43–47).—A colorimetric method for determining P in pharmaceutical products is described, based on the Bell and Doisy reaction given by phosphates with molybdic acid, quinol and Na_2SO_3. In addition to the estimation of P_2O_5 in phosphates, $PO_4^{\prime\prime\prime}$ can be determined as an impurity in organic compounds such as glycerophosphates, whilst the organic P is obtained after ashing the sample. The method can be applied to the estimation of hypophosphites after oxidation to $PO_4^{\prime\prime\prime}$ by fusion with KOH. J. H. WATON

899. **A spectrographic method for the determination of phosphorus in steels.** Le R. S. Brooks and F. R. Bryan (*Spectrochim. Acta*, 1954, **6** [5–6], 413–417).—A spectrophotographic procedure for determining from 0·003 to 0·061 per cent. of P in steels is described. The 2149·11 line of P is recorded on Ilford Q-2 plates requiring no special processing. The emulsion is calibrated by means of the Fe lines between 2100 and 2175 A. Use of a large Littrow quartz spectrograph with a 10-μ fixed entrance slit allows resolution of the 2149·11 line from the 2148·97 line of Cu. The precision and accuracy are adequate for routine purposes. W. J. BAKER

900. **Direct-reading spectrochemical determination of phosphorus in steel and iron-ore.** C. G. Carlsson and L. Danielsson (*Spectrochim. Acta*, 1954, **6** [5–6], 418–433).—The procedure described depends on the use of a quartz spectrograph (reciprocal dispersion $\simeq$ 5 A per min. at 2150 A) equipped with electron-multiplier tubes, a simple recorder, and a new slit arrangement with very precise setting of the exit-slits. A triggered low-tension a.c. spark ensures high sensitivity and precision, whilst more accurate values are obtained with a plane surface and C counter-electrode than with a pin sample. The reference line is Fe 2253·1 A, and the 2149·11 line of P is measured, although for high Cu contents the 2136·2 line of P is preferred. Contents of $>$ 0·005 per cent. of P can be determined in steel and iron-ore provided the Cu content is not above 0·4 per cent. The standard deviation is $< \pm$ 5 per cent., except for ores containing $<$ 0·1 per cent. of P for which the deviation is $\simeq$ 10 per cent. Such low-P ores cannot be fused before direct spectrometric analysis. W. J. BAKER

901. **Separation by paper chromatography of oxy-acids of phosphorus.** J.-P. Ebel (*Mikrochim. Acta*, 1954 [6], 679–700).—The oxy-acids of phosphorus can be separated almost completely by paper chromatography. Hypophosphite, phosphite, orthophosphate and hypophosphate can be separated by one-dimensional chromatography. The solution is spotted on to filter-paper (preferably Whatman No. 4) and dried at room temp. Ascending chromatography is used for development; acid or alkaline solvents may be used. For the former, good separations have been attained with *iso*propanol - trichloroacetic acid - aq. NH_3 soln., but ethanol - trichloroacetic acid - aq. NH_3 soln. or butanol - H_2O - picric acid - aq. NH_3 soln. are also satisfactory. The solvents differ in rapidity; the ethanol solvent is the most rapid, but gives spots that tend to spread into bands. The alkaline solvents are mixtures of alcohols with aq. NH_3 soln., the most successful being *iso*propanol - *iso*butanol - aq. NH_3 soln. For development, the oxy-acids are converted into molybdophosphates, which are then reduced by H_2S to molybdenum blue. For this method, the paper must be thoroughly washed with HCl, 8-hydroxyquinoline, ethanol and H_2O to remove traces of alkaline earths. The alternative method of development is to treat the paper successively with $FeCl_3$ and sulphosalicylic acid. The separation of the acids is easier if the method of two-dimensional chromatography is used. A preliminary separation is made by one-dimensional chromatography with one solvent. The paper is dried, turned through a right angle and a second chromatographic separation is carried out with another solvent. An alkaline solvent should be used first and an acid solvent second. In this method, the oxy-acids divide themselves into the following three groups: those acids with one atom of P, *viz.*, hypophosphite, phosphite and orthophosphate; hypophosphate and the linear polyphosphates, *viz.*, pyrophosphate, tripolyphosphate and Graham's salt; and the cyclic trimetaphosphate and tetrametaphosphate. Within each group, the separations are a function of the mol. wt., the substances moving less as the mol. wt. increases. The method can be used for quantitative analysis and has been applied to poly- and metaphosphates, but the error is high. A. J. MEE

902. **Analysis of phosphorus compounds. Automatic pH titration of soluble phosphates and their mixtures.** J. R. van Wazer, E. J. Griffith and J. F. McCullough (*Anal. Chem.*, 1954, **26** [11], 1755–1759).—The pH of a 100 to 150-ml sample of an aq. soln. containing 0·5 to 0·75 g of phosphate mixture (sodium ortho-, pyro- and polyphosphates only) is measured, and then sufficient *N* HCl is added to reduce the pH to $\simeq$ 3. This mixture is titrated potentiometrically (automatic titration pH meter) with 0·1 to 0·15 *N* NaOH; the titre between

the two inflexions at ≃ pH 4·5 and 9·5 is a measure of the weak acid concn. The pH of the titrated mixture is again adjusted to ≃ 3 with *N* HCl and the titration is repeated to just beyond the pH 4·5 inflexion; *N* $AgNO_3$ (50 ml) is added and the titration is continued to a new end-point at pH ≃ 5. This titre is a measure of the orthophosphate concn. Alternatively, this may be determined, if the PO_4''' concn. is high enough, by the addition of the *N* $AgNO_3$ after the end-point at pH ≃ 9·5 is reached, when the pH falls to ≃ 3, and completing the titration as above. A second sample is refluxed with 6 *N* HCl (10 ml) for 8 hr. to hydrolyse condensed phosphates, when the soln. is cooled and its pH adjusted to ≃ 3 with 6 *N* NaOH. Titration is carried out as above with 0·1 to 0·15 *N* NaOH. A slight modification for long-chain polyphosphates is described. From the results, by means of given formulae, the following percentages may be calculated: total P_2O_5, P_2O_5 as orthophosphate, P_2O_5 as end-groups, P_2O_5 as middle-groups, H_2O, Na_2O and average length of chain polyphosphates. All operations are claimed to have a precision of better than ± 0·5 per cent., except the orthophosphate determination, for which precision is ± 5 per cent.

D. A. PANTONY

903. **Determination of hypophosphite in the presence of phosphite.** D. N. Bernhart (*Anal. Chem.*, 1954, **26** [11], 1798–1799).—A sample containing approx. the equivalent of 0·05 g of NaH_2PO_2 or 0·1 g of NaH_2PO_3 is dissolved in dil. H_2SO_4 (1 + 4) (50 ml). To the soln is added Ce^{++++} [56 g of $2NH_4NO_3.Ce(NO_3)_4$ in 1 litre of dil. (1 + 10) H_2SO_4] (50 ml) and the mixture is either boiled for 15 min. (PO_2''' plus PO_3''' determinations) or allowed to stand at room temp. for 2 hr. or at 60° C for 30 min. (PO_2''' determinations). After cooling, the excess of Ce^{++++} are titrated with 0·1 *N* Fe^{++} (ferroin), the titre being a measure of either PO_2''' plus PO_3''' or PO_2''' only. Accuracy and precision are given as 2 parts per thousand.

D. A. PANTONY

904. **Analysis of mixtures of the condensed phosphates by ion-exchange chromatography.** S. Lindenbaum, T. V. Peters, jun., and W. Rieman, III (*Anal. Chim. Acta*, 1954, **11** [6], 530–537).—An earlier theoretical treatment (*Anal. Abstr.*, 1954, **1**, 1814) is extended to methods in which two or three eluting solutions are used to separate ortho-, pyro-, tri-, trimeta- and tetrametaphosphate ions on ion-exchange columns. With the aid of the derived equations a procedure is devised for the separation and determination of each of the five anions with a mean error of 0·2 per cent. of the sample.

W. C. JOHNSON

905. **Evaluation of commercial phosphates.** H. Étienne (*Ind. Chim. Belge*, 1954, **19** [10], 1021–1024).—Further information on the qual. and quant. analysis of phosphates in detergents (*Brit. Abstr. C*, 1953, 381) is given. Qual. tests for distinguishing between ortho-, pyro, tripoly-, tetrapoly- and polymetaphosphates are described, including the benzidine colour-test for orthophosphates and a number of specific reactions of phosphates with aq. hexa-amminocobaltic chloride. A slight modification of the method for determining total P_2O_5 by alkaline hydrolysis of the pptd. ammonium molybdophosphate gives greater sensitivity and accuracy. Conditions governing the quant. pptn. of barium phosphates at pH controlled between about 1·8 and 4 have been re-assessed; procedures for determining SO_4'', Cl', combined Na_2O and alkalinity are given. The two different crystalline forms of tripolyphosphate can be distinguished by the different rises of temp. of each on hydration.

W. J. BAKER

906. **A colorimetric determination of inorganic pyrophosphate.** R. M. Flynn, M. E. Jones and F. Lipmann (*J. Biol. Chem.*, 1954, **211** [2], 791–796).—A colorimetric method is described for the determination of 0·05 to 0·5 μ-mole of P_2O_7''''. It is based on a cysteine-catalysed colour reaction with the molybdate reagent of Fiske *et al.* (*Brit. Abstr. A*, 1926, 443). The sample containing 0·1 to 0·5 μ-mole of P_2O_7'''', and preferably less than 1 μ-mole of PO_4''', is put into a 10-ml volumetric tube. Water to approx. 7 ml, molybdate reagent (1·0 ml) and cysteine solution (0·4 ml, 70 μ-moles) are added to all the tubes. Then 0·4 ml of a solution of Eikonogen (1-amino-2-naphthol-4-sulphonic acid) in aq. $NaHSO_3$ - Na_2SO_3 is added, the vol. in each tube is adjusted to 10 ml with water and the contents are mixed immediately. After keeping at approx. 25° C for 7 min., the intensity of the colour is determined colorimetrically using filter No. 66; this gives the amount of PO_4'''. For small amounts of P_2O_7'''', the vol. may be reduced to 5 ml, or 1 ml if a Beckman spectrophotometer with micro-cells is available. As exact timing is very important for the determination of P_2O_7'''', additions of Eikonogen are spaced 0·5 to 1 min. apart. Readings are made at the same time-intervals after 7 and 90 min. exactly. The difference between these readings gives the amount of P_2O_7''''. With high concn. of PO_4''', a correction factor must be applied; a further correction is needed if adenosinepolyphosphates are present.

J. N. ASHLEY

907. **Separation of antimony from other metals by rapid electrolysis of sulphate solutions. II. Separation from arsenic, lead, iron and zinc. III. Separation from tin.** M. S. Jovanović (*Bull. Soc. Chim., Belgrade*, 1954, **19** [5], 289–295, 297–303).—**II.** Nitric acid is added to an aq. H_2SO_4 solution containing Sb and As to prevent the reduction of As^V to As^{III}, and the solution is electrolysed (30 min. at 80° C, at 1·8 to 2·0 V) until the current falls to 0·1 amp. The cathode is washed with H_2O and ethanol, dried at 80° to 90° C and weighed. Arsenic is determined as $Mg_2As_2O_7$ in the residual electrolyte. The electrolytic method is not applicable in the presence of Pb, as Pb - Sb alloys are deposited under all conditions studied. In the presence of Fe, conc. aq. NaOH is added until a ppt. appears, when 1 ml of conc. H_2SO_4 is added. The solution is electrolysed at 90° C, initially at 2·0 V and 1·2 to 1·4 amp., the voltage is lowered to 1·8 V, and the process is continued until the current falls to 0·2 to 0·4 amp., after which electrolysis is continued for 30 min. at 2·0 V; the cathode is weighed as before, and Fe is determined in the residual electrolyte as Fe_2O_3. In the presence of Zn, the solution is electrolysed at 80° C (2·4 amp., 2·2 V) until the ppt. disappears, when the voltage is lowered to 1·8; electrolysis is continued until the current falls to 0·1 to 0·2 amp., when it is further continued for 30 min. at 2·2 V, the current falling from 1 to 0·3 amp., after which the cathode is washed, dried, and weighed as before. Excess of NaOH is then added to the electrolyte, and Zn is determined electrolytically. **III.** Sb - Sn alloy is dissolved in 10 ml of conc. H_2SO_4, 3 ml of conc. HNO_3 are added to the cooled solution, followed by 100 ml of H_2O, 10 g of Na K tartrate, and conc. aq. NaOH until the ppt. is dissolved. The solution is made neutral to litmus with H_2SO_4.

and electrolysed (80° C; initially 1 amp. and 2·4 V falling to 0·1 amp. and 2·2 V). One ml of 50 per cent. H_2SO_4 is added, and electrolysis is continued for 30 min. at 2·7 V, after which the deposit of Sb is weighed. Two ml of 50 per cent. H_2SO_4, 10 g of oxalic acid and 5 g of hydrazine are then added to the residual electrolyte and the solution is electrolysed (80° C; 3 amp. for 30 min.); with the same antimony-coated cathode, further portions of oxalic acid and hydrazine are added, and electrolysis is continued for a further 30 min., after which the deposit of Sn is weighed. The results are about 0·2 per cent. greater than those found by the usual volumetric procedure for Sn. The method is not applicable in the presence of Cu or Pb.

R. Truscoe

908. **The use of a mixture of 8-hydroxyquinoline hydrochloride and potassium iodide for the detection of antimony in the presence of tin.** S. M. Éfros (*Tr. Leningr. Tekhn. In-ta*, 1953, [27], 116–118; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 29,318).—A soln. of oxine and KI in 6 to 7 *N* HCl gives a ppt. (probably $C_9H_7ON.HSbI_4$) with Sb^{III}; the ppt. dissolves in water on heating. Sn^{II} or Sn^{IV} do not interfere. The reagent may be used to detect Sb^{V}; the KI reduces Sb^{V} to Sb^{III} and the liberated iodine is removed by the addition of $SnCl_2$ or Na_2SO_3; AsO_4''' does not interfere if present. The minimum amount of Sb detected is 2 μg. Cations of the analytical group 4 must be removed before the test is carried out. F. Hayes

909. **New method of colorimetric determination of a small quantity of vanadium by means of formaldoxime.** M. Tanaka (*Mikrochim. Acta*, 1954, [6], 701–707).—Formaldoxime gives a yellow colour with V^{IV} compounds which is more intense than any other colour formed with V. The reaction occurs in alkaline (ammoniacal) solution. The stability of the colour varies with temp.; between 10° and 20° C the coloration reaches a max. in 15 min. and does not decrease over a period of 24 hr. The effect of interfering ions is studied; Fe, Co and Ni ions must be removed before carrying out the procedure.

A. J. Mee

910. **Analysis for industry.** [**Separation of niobium, tantalum and zirconium.**] T. S. West (*Ind. Chem.*, 1954, **30**, 550–552).—Recent work on the analytical separation of Nb from Ta or Zr in mineral acid solution by means of org. solvents is summarised. Nb is quantitatively extracted from solutions in 9·6 *M* HCl by a solution of methyldioctylamine in xylene, 1·4 per cent of Ta being extracted at the same time. Ta is preferentially (80 to 95 per cent.) extracted by di*iso*propyl ketone from mixtures of HF with HCl, HNO_3, H_2SO_4 or $HClO_4$, 0·43 to 11 per cent. of Nb also being extracted. Better separation is attained from HF - HCl solutions by means of a ketone such as *iso*butyl methyl ketone. Similar methods of extracting Nb and Zr are also discussed. (13 references.)

D. R. Peck

911. **The elimination of the blank value in the Unterzaucher method for the micro-determination of oxygen.** A. F. Colson (*Analyst*, 1954, **79**, 784–785).—On the assumption that the persistent blank value obtained by several workers in Unterzaucher's method for the micro-determination of O was due to the incomplete removal of air adsorbed on the carbon black and on the inner surface of the combustion tube, a silica combustion tube packed in the usual way with 4 cm of silica chips, 14 cm of pelleted carbon and 1 cm of silica wool was gently tapped until a clear passage was left above the carbon pellets. The exit end was then closed and the section containing the carbon was heated at 1120° C for 2 hr, the tube being continuously evacuated by a Hyvac pump. The unpacked portion of the tube was heated similarly for 1 hr. and the C heated again for 2 hr. After re-admission of N and re-adjustment of the C to its normal position, a stream of N was passed through the whole assembly for 24 hr., the furnace temp. being maintained at 1120° C, and the anhydro-iodic acid was heated to 118° C. When 180 ml of N (9 ml per min.) were passed through the apparatus, no measurable blank was observed in the 30 to 40 tests made during the working life of the tube. A. O. Jones

912. **Simple objective method for estimating low concentrations of ozone in air.** J. R. Beatty and A. E. Juve (*Rubb. World*, 1954, **131** [2], 232–238).—The device described measures the differential creep between two halves of a rubber thread, one half being immersed in the atmosphere to be tested, the other being shrouded. The ends of the thread are fixed, and the remainder passes round a free pulley to which a pointer moving over a scale is attached. Attack by ozone on the exposed half causes cracking of the thread with resulting increase of strain and rotation of the pointer. Rate of pointer-deflection is linearly dependent on ozone concentration. A deflection of 23° was obtained at 18° C in 30 min. with 13 p.p.m. of ozone in air. The thread used is a natural-rubber golf-ball thread 0·075 in. × 0·015 in. at 100 per cent. elongation. The effects of experimental variables and ozone concentration are stated and experimental results on the use of the instrument to measure ozone concentration in the atmosphere and the resistance of gum stocks of diverse formulation to ozone are tabulated.

J. L. Prosser

913. **Micro-determination of sulphur in metals, slags, chromium baths, organic substances and gases.** G. Graus with A. Zöhler (*Angew. Chem.*, 1954, **66** [15], 437–442).—Very small amounts of S are rapidly and accurately determined by the electrolytic - potentiometric titration of H_2SO_4. This H_2SO_4 is formed by the absorption in dil. H_2O_2 of the SO_2 formed by burning sulphur-containing materials in a stream of oxygen. Generally the substance is mixed with CuO before combustion. Variations in the technique for determining small amounts of S in W and Mo and their oxides and for substances with which the method cannot be applied directly are given. For the last-named substances, the decomposition is effected by the Carius and Wurzschmitt procedure and the $BaSO_4$ is co-pptd. with $BaCrO_4$ or $Fe(OH)_3$, the ppt. then being decomposed in a melt of $Ca_3(PO_4)_2$ and CuO in the combustion process. H. Wren

914. **A differential alkalimetric determination of sulphuric acid - hydrochloric acid and sulphuric acid - nitric acid mixtures.** F. E. Critchfield and J. B. Johnson (*Anal. Chem.*, 1954, **26** [11], 1803–1806).—H_2SO_4 - HCl and H_2SO_4 - HNO_3 mixtures are titrated in methyl cyanide with standard morpholine reagent by one of the five following methods. (*a*) In this method, which is recommended when small quantities of weak organic acids are present, 75 ml of methyl cyanide are carefully neutralised with aq. 0·1 *N* HCl (screened methyl yellow), 2 to 5 milli-equiv. of total acid are added and the soln. is titrated potentiometrically with 0·1 *N* morpholine in methyl cyanide. (*b*) After the first

inflexion in the titration curve of method (*a*), 75 ml of water are added and the titration is completed as above. (*c*) One-hundred ml of methyl cyanide are neutralised as above, 8 to 20 milliequiv. of total acid are then added and the soln. is made up to 200 ml; a 50-ml aliquot is titrated with 0·1 *N* morpholine to the first green end-point (screened methyl yellow), which corresponds to the first potentiometric inflexion. A second 50-ml aliquot is titrated to the green end-point of bromocresol green, which corresponds to the second potentiometric inflexion. (*d*) This method is similar to method (*b*) but the titration medium is a (3 + 1, by vol.) mixture of methyl cyanide and methanol, neutralised as above, and 0·5 *N* morpholine in methyl cyanide is used as titrant. (*e*) This again is similar to method (*c*), but the titration medium and titrant are as for method (*d*). The first end-point corresponds to the neutralisation of half the H_2SO_4 plus the HNO_3 (or HCl), and the second to the remaining half of the H_2SO_4. Results on synthetic mixtures show that > 99 per cent. of the added acids are accounted for. Other acids, *e.g.*, H_3PO_4, $HClO_4$ and toluenesulphonic acid, interfere, except when small quantities of weak organic acids, such as ⩾ 2 per cent. of acetic acid, are present in method (*a*). D. A. PANTONY

915. **The preparation of standard solutions of sulphuric acid by means of specific gravity determinations.** R. E. Essery (*Anal. Chim. Acta*, 1954, **11** [6], 501–506).—In the range 32 to 39 per cent. w/w, the percentage (*P*) by wt. of H_2SO_4 is related to the sp. gr. at 20° C/20° C according to the equation $P = 343{\cdot}61\ (\log_{10}\text{sp. gr.}) + 0{\cdot}1875$. These H_2SO_4 solutions are not hygroscopic and *N* H_2SO_4 can be prepared from them by weighing (4901·6 ÷ *P*)g and diluting to 1 litre. The system is based on weights in air. In the sp. gr. determination, an error of 0·1° C causes an error of ≃ 0·009 per cent. in the concn. W. C. JOHNSON

916. **Indirect polarographic determination of sulphates. II. Application to the analysis of waters.** O. A. Ohlweiler (*Anal. Chim. Acta*, 1954, **11** [6], 590–593).—The method described is based on previous studies of the system $[SO_4'']/[Pb^{\cdot\cdot}]/[PbSO_4]$ (*Anal. Chim. Acta*, 1953, **9**, 476). *Procedure*—Remove the cations present by passing the sample of water through a column of Amberlite IR-120 resin. Evaporate 50 ml of the eluate to dryness on a water-bath with 5 ml of 0·5 *M* KNO_3, dissolve the residue in water, evaporate the soln. to dryness and heat the residue at 120° C for 30 min. This treatment removes volatile acids. Dissolve the residue in 0·005 *M* HNO_3 containing 0·01 per cent. of gelatin, add 0·1 g of $PbSO_4$, bubble nitrogen through the soln. for 30 min. and determine the height of the polarographic lead wave. Prepare a calibration curve from known amounts of 0·005 *M* H_2SO_4 submitted to the same procedure, and plot wave heights against concn. of SO_4''. Concn. of $0{\cdot}5 \times 10^{-3}$ to $1{\cdot}5 \times 10^{-3}$ *M* of SO_4'' provide the best operating conditions. The max. error is < 3 per cent. W. C. JOHNSON

917. **Oxidation with alkaline permanganate using formic acid for the back-titration. I. Determination of quadrivalent tellurium and the redox potential of the $TeO_4'' - TeO_3''$ system.** I. M. Issa, R. M. Issa and A. A. Abdul Azim (*Anal. Chim. Acta*, 1954, **11** [6], 512–523).—Tellurite solutions (0·02 to 0·09 *N*) can be titrated potentiometrically with 0·005 to 0·02 *N* $KMnO_4$ in the presence of 0·1 to 4 *N* NaOH; the $KMnO_4$ is reduced to MnO_2. With weaker solutions, 0·00002 to 0·00046 *N* TeO_3'' and 0·00004 to 0·002 *N* $KMnO_4$, in the presence of 3·5*N* NaOH almost quant. reduction of MnO_4' to MnO_4'' occurs. Titrations can also be carried out in 0·1 *N* NaOH and in the presence of added telluric acid, and the MnO_2 then remains in solution. When, under otherwise similar conditions, $KMnO_4$ is titrated with TeO_2'', a mixture of reduction products is obtained, but the reaction is approx. quant. in the presence of added telluric acid. Tellurite can also be determined by adding the soln. slowly to a 100 per cent. excess of 0·1 *N* $KMnO_4$ in the presence of 1·5 to 2 *N* NaOH and titrating the excess of $KMnO_4$ with standard formic acid soln. The formal redox potential of the $TeO_4'' - TeO_3''$ system for 0·02 to 0·09 *N* TeO_3'' at unit OH' concn., calculated from the $KMnO_4$ titration curve, is ≃ 0·26 V. W. C. JOHNSON

918. **Determination of ethylenediaminetetra-acetic acid as the chromium complex.** P. J. Cherney, B. Crafts, H. H. Hagermoser, A. J. Boule, R. Harbin and B. Zak (*Anal. Chem.*, 1954, **26** [11], 1806–1809).—A method for the determination of ethylenediaminetetra-acetic acid (**I**) in urine, as the Cr complex, is described. *Procedure*—Two ml of urine are treated with 2 ml of 0·5 per cent. Na_2CrO_4 and 2 ml of 0·2 *N* arsenious acid [*sic*]. The soln. is then heated at 100° C for 5 min. and the absorbance is measured at 560mμ, the **I** content being calculated from a previously prepared calibration curve. Other constituents of the urine do not interfere, owing to the high formation constant of the Cr. Recoveries of added **I** from urine over the range 50 to 300 mg per 100 ml of urine were between 93 and 105 per cent. G. P. COOK

919. **A modified method for the decomposition of chromite.** P. D. Malhotra (*Analyst*, 1954, **79**, 785–786).—In an investigation on the composition of Indian chromites, various methods for the initial decomposition of the mineral were tried; fusion with Na_2CO_3 in an atmosphere of O proved the most rapid, effective and convenient method. *Procedure*—The chromite (0·5 g) in a platinum crucible is mixed with 4 to 5 g of anhydrous Na_2CO_3 and covered with a further 1 g. The crucible is heated on a Meker burner and when the melt is tranquil a stream of O is allowed to impinge on its surface by means of a bent platinum tube. Alternatively, a platinum Rose crucible may be used. The melt is swirled occasionally. The cooled melt is treated with water and the slurry is filtered. Cr, Al, Va and Si are in the filtrate and the residue containing Fe, Ca, Mg, Mn, Ti, etc., is completely sol. in dil. HCl. The platinum crucible is not attacked. A. O. JONES

920. **Chromatography and determination of tungsten.** A. Lacourt and G. Sommereyns (*Mikrochim. Acta*, 1954, [6], 604–629).—Two methods have been used for the chromatographic separation and determination of W occurring either alone or in the presence of Cr, V, Mo and Fe. In ordinary partition chromatography, the colour is developed directly without extraction of the W from the paper. The most favourable conditions for the qual. separation of the five elements in KCl solution require the use of a saturating solution composed of a mixture of pentanol (60 ml), benzene (10 ml) and conc. HCl (30 ml), and a developer composed of pentanol (45 ml) and 85 per cent. formic acid (55 ml). After elution for 2 hr. with 2 per cent. KCl, the liquid and washing water are evaporated to dryness. The

residue is rapidly cooled and 3 drops of conc. HCl and 1 ml of an 0·2 per cent. w/v solution of Dithiol in butyl acetate are added. The mixture is heated for exactly 30 min. on a water-bath. After rapid cooling 10 ml of butyl acetate are added. After 15 min. the intensity of the coloration is determined on a Beckman spectrophotometer at 630 mμ. Reproducibility is ± 2 to 3 per cent. for 10 μg. In the second method, different solvents are used for saturation and development. Under these conditions, reproducibility can be as good if the process is carried out as described; by the usual method results for W are as much as 14 per cent. too low. The interference of other metals is considered. With a KCl solution containing W, Cr, Mo, V and Fe, Cr is without effect, but Mo causes a marked increase in the optical density. If the W is first separated from these elements by chromatography, the presence of V in the eluting solution does not affect the results of the spectrophotometric determination of W. A. J. MEE

921. **Methods of the analysis of iron and steel. XXXII. Tungsten in steel. (Absorptiometric method.)** British Standards Institution (B.S. 1121: Part 32, 1954, 7 pp.).—This absorptiometric method depends on measurement of the colour of the tungsten-thiocyanate complex at ≃ 430 mμ. *Procedure*—One g of sample is dissolved in 40 ml of a solution containing 15 ml of phosphoric acid (sp. gr. 1·75) and 15 ml of H_2SO_4 (sp. gr. 1·84) per 100 ml, 6 ml of phosphoric acid (sp. gr. 1·75) are added and the solution is oxidised with nitric acid and fumed. A water extract is filtered and diluted to 100 ml and from this two 5-ml portions are taken. To one portion, 40 ml of 0·5 per cent. stannous chloride, 3 ml of 25 per cent. ammonium thiocyanate and 1 ml of 2·5 per cent. titanous chloride solution are added, with mixing between each addition; to the other, 40 ml of 0·5 per cent. stannous chloride only are added. Both are diluted to 50 ml. The difference between the absorptions of the two solutions (Ilford 601 filter with the Hilger photo-electric absorptiometer) is evaluated as percentage tungsten from a standard graph; a correction for vanadium is made if necessary. J. O. LAY

922. **Stability of dilute solutions of uranium, lead and thorium ions.** R. G. Milkey (*Anal. Chem.*, 1954, **26** [11], 1800–1803).—Nitrate solutions containing $Pb^{\cdot\cdot}$, $UO_2^{\cdot\cdot}$ and $Th^{\cdot\cdot\cdot\cdot}$ (0·1 to 1000 μg per ml) were stored in borosilicate glass and polythene containers for ≃ 10 weeks. Concn. were then determined by standard methods ($Pb^{\cdot\cdot}$ by dithizone colour, $UO_2^{\cdot\cdot}$ fluorimetrically and $Th^{\cdot\cdot\cdot\cdot}$ by alizarin S or 1-*o*-arsonophenylazo-2-naphthol-3;6-disulphonic acid colours). Solute losses were apparently similar in both glass and plastic containers, and are not recovered by prolonged shaking. Adsorption and hydrolysis account for most of the solute losses, especially as serious losses occur only when the pH of the soln. is greater than the pH at which hydrolysis commences (*viz.*, $Pb^{\cdot\cdot}$ at > 5·6, $Th^{\cdot\cdot\cdot\cdot}$ at > 3·0, $UO_2^{\cdot\cdot}$ at > 4·2). Values of solubility products are calculated from the observations and compared with published values. D. A. PANTONY

923. **Polarographic determination of uranium in ores in ascorbic acid supporting electrolyte.** M. Šušić, I. Gal and E. Cuker (*Anal. Chim. Acta*, 1954, **11** [6], 586–589).—Samples (1 to 2 g) of uranium ores, containing 0·01 to 0·1 per cent. of U, are treated with such solvents as conc. HNO_3 and aq. HF (40 per cent.), H_2SO_4 is added and the solutions are evaporated to dryness. After the residue has been dissolved in water, ascorbic acid is added to produce a concn. of 0·5 *M* in the final soln., the pH is adjusted to 3·6 to 4·0 with aq. NH_3, 0·2 to 0·4 ml of aq. gelatin (1 per cent.) is added, the soln. is diluted to 25 or 50 ml and the U content is determined polarographically. The U wave occurs at −0·35 to −0·45 V *vs.* the S.C.E. and the wave height is converted to U content by the method of standard addition. For ores containing 0·01 to 0·05 per cent. of U, the relative error is 9 to 5 per cent.; higher contents can be determined more accurately. Te, Tl and Pb interfere but Fe, V, Mo, Cr and Ti and small amounts of Cu are without effect. W. C. JOHNSON

924. **Analysis of halide mixtures by ion-exchange chromatography.** R. C. DeGeiso, W. Rieman, III, and S. Lindenbaum (*Anal. Chem.*, 1954, **26** [11] 1840–1841).—A halide sample containing ≯ 2·6 milli-equiv. of any one halide are dissolved in 0·5 *M* $NaNO_3$ (2 ml) and the soln. is poured through a 6·7 cm × 3·4 sq. cm anion-exchange column (Dowex 1-X10, 100 to 200 mesh; see *Brit. Abstr. C*, 1952, 542) that has been equilibrated with 0·5 *M* $NaNO_3$. The column is developed with the same solvent at a flow rate of 1 cm per min.; Cl' appears in the first 55 ml of eluate. Then 2 *M* $NaNO_3$ is run through at the same rate; the first 55 ml of this eluate contains Br', and the I' appears quant. in the third fraction of 260 ml. The Cl' (0·05 *N* $Ag^{\cdot}$), Br' (0·05 *N* $Ag^{\cdot}$) and I' (0·05 *N* $Ce^{\cdot\cdot\cdot\cdot}$) are determined by standard potentiometric titration methods. Average error is given as ± 0·09 per cent. of the total halide for any one halide. The method responds to the analysis of traces of any one halide in the presence of a large excess of the other two; average error is ± 0·02 per cent. of total halide. D. A. PANTONY

925. **The determination of potentially ionic fluorine in non-aqueous solvents.** H. F. Liddell (*Analyst*, 1954, **79**, 752–754).—The thorium lake of Chromotrope 2B (Colour Index No. 45) forms a bluish-violet dispersion in ethanol containing ethylcellulose. In ethanol or other organic solvents this colour is changed to the intense red of the original dye in the presence of potentially ionic F and 0·02 p.p.m. of this ion can thus be detected. The change of colour can be measured absorptiometrically at 515 mμ or, at great dilution, visually in Nessler cylinders. Alternatively, with toluene as the main solvent the unchanged lake can be pptd. with "triethylamine phthalate," removed by filtration and the red colour alone observed. The diphenylguanidine salt of Chromotrope 2B (prep. described) is more sol. in ethanol and is used for the preparation of the stock soln. of the dye. Precision is not high, but the sensitivity of the method and the absence of interference by substances insol. in the organic solvents make it of use. A. O. JONES

926. **Polarographic determination of manganese in air.** I. B. Kogan and S. L. Makhover (*Gigiena i Sanit.*, 1954, [2], 52–53; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 29,321).—Manganese (2 to 5 μg) is absorbed by hygroscopic wool in a dust-tube; the wool is incinerated at 600° C in a muffle furnace and the residue is dissolved in 1 ml of conc. HCl. The acid is removed by evaporation on a water-bath and the residue is dissolved in a little water. The soln. is transferred to a polarographic cell, a drop of 0·15 per cent. gelatin soln. and 2 ml of 0·2 *N* KCNS soln. are added, and the soln. is polarographed at a sensitivity of 1/25. The Mn half-wave potential is − 1·55 V (*vs.* the S.C.E.). E. HAYES

927. **Determination of trace amounts of iron, nickel and vanadium by fluorescent X-ray spectrography.** G. V. Dyroff and P. Skiba (*Anal. Chem.*, 1954, **26** [11], 1774–1778).—A finely ground sample of a cracking-catalyst is packed into the holder of an X-ray spectrograph. The sample is subjected to X-ray bombardment in the standard manner (tungsten target), the emissions being analysed in a crystal of LiF and a helium path being used to minimise atmospheric absorption. The elemental voltage and current, wavelength of emissions and angle (2θ) of measurement of emission and background are as follows: $Ni_{K\alpha}$, 35 kV, 20 mA; 1·66 A; 48·48°, 54°. $Fe_{K\alpha}$, 35 kV, 20 mA; 1·94 A; 57·20°, 54°. $V_{K\alpha}$, 50 kV, 50 mA; 2·50 A; 76·70°, 74°. The intensity of emission (counting rate) is compared with that of standards, and in the calculation of concn., allowance is made for blank runs (impurities in the X-ray tube). The following standard deviations are given: Fe, 3 per cent. (relative), Ni, 0·002 per cent. and V, 0·002 per cent. over the ranges 0·1 to 1, 0·002 to 0·1 and 0·002 to 0·1 per cent., respectively. D. A. PANTONY

928. **Micro-analysis as an important method of investigation in the development of steel.** W. Koch (*Mikrochim. Acta*, 1954, [6], 760–781).—Micro-analytical methods must be used in the determination of the structural constituents of steel, because the quantities of the various phases that can be liberated from the structure of the steel do not exceed 1 to 15 mg. A. J. MEE

929. **Quantitative chromatographic separation on paper of cobalt, copper and zinc in solution.** A. Lacourt and P. Heyndryckx (*Mikrochim. Acta*, 1954, [6], 630–647).—The chromatographic separation of Co, Cu and Zn has been extensively studied. The saturating solution and developer were ethyl methyl ketone, pure or aqueous. The mixture contained 10 μg per 0·01 ml of $CoCl_2$, $CuCl_2$ and $ZnCl_2$ in dil. HCl (1 + 1). The effects of the type of solvent, the paper, the contaminant and the concn. of the solutions on the dimensions of the spots were investigated. Standard conditions for carrying out the separation are given. Under these conditions, 0·3 μg of Co and 0·1 μg of Cu can be determined. For these low concn., at which the stains are almost invisible to the eye, the photometric method is used to detect and determine the elements. A. J. MEE

930. **Chelation of platinum-group metals. Spectrophotometric determination of palladium with ethylenediaminetetra-acetic acid.** W. M. MacNevin and O. H. Kriege (*Anal. Chem.*, 1954, **26** [11], 1768–1770).—A soln. containing platinum-group metals is treated with dimethylglyoxime (see *Brit. Abstr. C*, 1953, 424) to precipitate Pd, and the Pd-dimethylglyoxime ppt. is dissolved in aqua regia. After evaporation of the soln. to dryness, the residue is taken up in 0·1 *M* HCl, and an excess of disodium ethylenediaminetetra-acetate is added. The pH and palladium concn. are adjusted to either 7 ± 1 (9 to 90 p.p.m.) or 1·8 ± 0·2 (18 to 180 p.p.m.) and the absorption is measured at 337 or 377 mμ, respectively. Pd concn. is calculated from standards. The dimethylglyoxime separation is avoided in the absence of other platinum-group metals. The change of absorption per pH unit is found to be 0·012 (pH range 5 to 9) and 0·032 (pH range 1·6 to 2·0), and this relationship is suggested as a basis for accurate determinations of Pd. Analytical figures for six determinations are given. The Beer-Lambert law holds over the range 5 to 200 p.p.m.; the colour develops rapidly and is stable. D. A. PANTONY

See also Abstracts **827, 834, 836, 837, 840, 841, 967, 968, 969, 1038, 1057, 1058, 1059, 1060, 1069, 1070, 1073, 1074, 1077, 1085.**

3.—ORGANIC ANALYSIS

931. **Wet carbon combustion and some of its applications.** D. D. van Slyke (*Anal. Chem.*, 1954, **26** [11], 1706–1712).—A partial review of the development of wet carbon combustion is presented. The use of a mixture of iodic and chromic acids in H_2SO_4 and H_3PO_4 to effect complete combustion in 1 or 2 min., is discussed; methods include the determination of the evolved CO_2 by manometric measurement, by weighing as CO_2 or $BaCO_3$, or by titration. Application of the method to the determination of fats, proteins and various elements pptd. with organic precipitants and to radioactive-carbon determination is also given. G. P. COOK

932. **Use of vanadium pentoxide in micro-determination of carbon and hydrogen.** S. Hirooka (*J. Chem. Soc. Japan*, 1954, **75** [2], 236).—Carefully prepared V_2O_5 can be used as the main filling in the combustion tube instead of Pt. Purified NH_4VO_3 (11 g) is suspended in water and V_2O_5 is pptd. by HCl. Well-washed pumice stone (10 to 14 mesh, 10 g) is mixed with the wetted V_2O_5, and the mixture is dried in a crucible and heated to dull redness. This procedure is repeated until all the V_2O_5 is fixed on pumice. The pumice is then sieved and put in the combustion tube. Analytical procedures can be carried out as usual. At a furnace temp. of 750° C, V_2O_5, which melts at 660° C, remains on the pumice without flowing out and attacking the tube. When air is used instead of oxygen (*Mikrochem.*, 1952, **39**, 166), the results are generally good, except for samples not easily charred (furnace temp., 800° C). K. SAITO

933. **Rapid methods of micro-elementary analysis. VIII. Simultaneous micro-determination of carbon, hydrogen and phosphorus in organic phosphorus compounds containing C, H, O, P and N.** M. O. Korshun, Ev. A. Terent'eva and V. A. Klimova (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 275–281).—In a modification of the combustion method for C and H, P is oxidised to P_2O_5 on pumice and then weighed. G. S. SMITH

934. **A method of determination of sulphur-35 in organic sulphur compounds.** S. Ikeda (*J. Chem. Soc. Japan*, 1954, **75** [1], 97).—A combustion method is used instead of the conventional Carius method for the measurement of ^{35}S contents in organic substances. The combustion tube is slightly modified to be suitable for the removal of the last traces of SO_3 and H_2SO_4. The total amount of S can be found by titration with NaOH. Sulphate ions are precipitated as $BaSO_4$ and the radioactivity is measured. The most favourable amount of $BaSO_4$ for the activity measurement is 30 to 60 mg. This method is especially suitable for organic sulphides. K. SAITO

935. **The propionyl index and its determination.** M. Pesez (*Bull. Soc. Chim. France*, 1954, [10], 1237–1238).—The sample of alcohol or amine (0·5 to 1·5 millimole) is placed in a 5-ml conical flask, which is stoppered after the contents have been treated with 2 ml of a reagent made by dissolving toluene-*p*-sulphonic acid (1 g) (free from H_2SO_4) in acetic acid

(30 ml), adding propionic anhydride (5 ml) and setting aside for 15 min. The mixture is stirred and kept at room temp. for 2 hr., or at 100° C for 30 min., and then cooled for 15 min. The contents of the flask are then added to 25 ml of 0·1 *M* aniline in benzene with 30 ml of acetic acid. After 5 min., the solution is titrated with 0·1 *N* $HClO_4$ in acetic acid to an emerald-green end-point with crystal violet. A correction is given for the difference between the temp. during titration and standardisation and for the presence of water (separately determined). A list of calculated and experimental indices for 38 alcohols and phenols and for 8 amines is given. E. J. H. BIRCH

936. **Application of infra-red spectrometry to the analysis of combustion products containing carbon dioxide, carbon monoxide and small proportions of propane.** A. P. C. Cumming (*J. Appl. Chem.*, 1954, **4** [10], 561–568).—Calibration curves of optical densities plotted against concentrations at wavelengths of 14·98, 4·65 and 3·3 μ for the gases carbon dioxide, carbon monoxide and propane, with nitrogen as diluent, are given. Deviations from Beer's law are apparent and errors occur in analysing mixtures of the gases. Correction curves are given that allow accuracies in analysis of within 0·1, 0·2 and 0·4 per cent. for propane, carbon dioxide and carbon monoxide, respectively. I. JONES

937. **Analysis of aqueous mixtures of hydrogen peroxide and aldehydes.** C. N. Satterfield, R. E. Wilson, R. M. Leclair and R. C. Reid (*Anal. Chem.*, 1954, **26** [11], 1792–1797).—Several methods were evaluated for the determination of total peroxide, total aldehyde and formaldehyde in aqueous mixtures. Iodimetric titrations were used for the determination of total peroxide; results were best by the method involving iodine liberation with HI - glacial acetic acid mixture, recoveries of > 99·7 per cent being attained. For the determination of total aldehyde, oxidation by alkali - H_2O_2 mixture was preferable to acid liberation with hydroxylamine hydrochloride, recoveries of between 98·5 and 101·0 per cent. being attained over the concn. range of 1·78 to 8·67 per cent. of aldehyde in the solution. For the determination of formaldehyde, colorimetric determination with Schiff's reagent, after reduction of the peroxides, gave the best results; the error was within ± 3 per cent. over the concn. range 2·26 to 28·0 per cent. of formaldehyde in the soln. G. P. COOK

938. **Studies on enol titration. I.** A. Gero (*J. Org. Chem.*, 1954, **19** [3], 469–471).—The original form of the Kurt Meyer titration of the enol content of a ketone with Br (*Ann.*, 1911, **380**, 212) has been improved by the use of ICl instead of Br. The ketone (containing about 0·1 millimole of enol) is mixed with 0·1 millimole of dry $NaHCO_3$: a 2·5 *N* soln. of ICl in methanol (0·1 ml) is added, followed immediately by 2 to 3 ml of a 15 per cent. w/v aq. NaI soln., and the liberated I is titrated. The $NaHCO_3$ prevents the acid formed liberating more enol. N. E.

939. **The separation of mixtures of methyl ketones.** N. J. Berridge and J. D. Watts (*J. Sci. Food Agric.*, 1954, **5** [9], 417–421).—Gas - liquid chromatography has been applied to the separation of mixtures of methyl ketones. A V-shaped column, packed with kieselguhr mixed with silicone, was placed in an oil-bath, whose temperature could be made to rise at a controlled rate of 0·17° C per min. Up to 1 ml of ketones in anhydrous ethanol or *iso*pentane was added to a reservoir at the entrance of the column through which the gas stream also passed. The gas flow was adjusted to 10 to 12 ml per min. at the exit and the effluent gas was allowed to interact with a continuous stream of dilute hydroxylamine hydrochloride. The fraction collector was emptied hourly and the pH values and volume of reagent in each fraction were recorded and compared with a freshly prepared standard graph from which the total ketone content of each band was calculated. This method was more accurate than continuous titration with the reagent. D. LIFF

940. **The absorptiometric determination of acetylacetone with ferric iron.** T. G. Bonner and M. Thorne (*Analyst*, 1954, **79**, 759–761).—An absorptiometric method is described for the determination of acetylacetone in aq. soln. by converting it to the sol. red ferric salt. *Procedure*—To 5 ml of a 6·5 per cent. w/v soln. of ferric ammonium sulphate in 0·1 *N* H_2SO_4, a measured vol. of the acetylacetone soln. (which must be 0·1 *N* with respect to H_2SO_4) is added and then 0·1 *N* H_2SO_4 until the vol. is 20 ml. After 15 min., the colour is measured in a Spekker absorptiometer with an Ilford No. 603 blue-green filter against an equal vol. of 0·1 *N* H_2SO_4. The influence of acidity and Na_2SO_4 on the intensity of the colour was investigated. The method was shown to be applicable to a Schiff's base of acetylacetone. A. O. JONES

941. **The paper chromatography of aliphatic dicarboxylic acids.** H. Kalbe (*Hoppe-Seyl. Z.*, 1954, **297** [1], 19–44).—A paper-chromatographic method for separating normal and branched-chain C_2 to C_{12} aliphatic acids has been worked out. Seven satisfactory solvent mixtures are given: (*i*) xylene - phenol - 85 per cent. formic acid (5:5:2 by wt.), (*ii*) as (*i*) but with ratios 7:3:1, (*iii*) *iso*pentyl formate - water - 98 to 100 per cent. formic acid (11:1:2 by vol.), (*iv*) *n*-pentyl formate - water - 98 to 100 per cent. formic acid (7:1:2 by vol.), (*v*) *iso*propanol - 1·5 *N* aq. ammonium carbonate in 3 *N* aq. ammonia (3:1 by vol.), (*vi*) as (*v*) with the substitution of *n*-propanol for *iso*propanol, (vii) 3 *N* tetrahydrofuran - 1·5 *N* aq. ammonium carbonate in 3 *N* aq. ammonia (4:1 by vol.). Mixtures (i) to (*iv*) are useful for separating the lower acids, and the basic ones for separating the others. Instead of using R_F values, sebacic acid, which has the highest R_F value of the readily available acids, was always run and the R_S value was used. The R_S value, which is the ratio of R_F of the unknown to the R_F of sebacic acid, increases with an increase in the number of carbon atoms; this ratio can be used to fix the mol. wt. of an unknown acid. A 0·03 per cent. soln. of methyl red in 0·05 *M* borate buffer at pH 8·0 is sensitive for identifying the position of the acids; 1 to 2 μg can be detected. The construction of an air-tight glass chamber in which both ascending and descending chromatograms may be run is described. F. POWELL

942. **Colour reaction of complex formation of elaeostearic acid with picric acid and a qualitative test for tung oil.** V. P. Goguadze and T. A. Pkheidze (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 308–313).—Picric acid in glacial acetic acid gives an intense-red colour with elaeostearic acid. A colour reaction with picric acid appears to be restricted to the aromatic series, apart from this one example. The limit of detection is 91 mg in 2 ml. The reaction can be used to detect ≮ 1 per cent. of tung oil in a mixture with other oils. G. S. SMITH

943. **The conversion and characterisation of sugars with sulphonylhydrazides.** O. Westphal, H. Feier, O. Luderitz and I. Fromme (*Biochem. Z.*, 1954, **326** [2], 139–149).—The methods of preparation of several sulphonylhydrazides and of the corresponding sulphonylhydrazones of the monosaccharides are reported, together with tables of melting points and other information for the characterisation of such derivatives.
G. W. CAMBRIDGE

944. **Polarographic reduction of dibromoethane and 1 : 2-dibromo-1-chloroethane, the bromination products of ethylene and vinyl chloride.** A. V. Ryabov, G. D. Panova and L. I. Efimov (*Uch. Zap. Gor'kovskogo Gos. Un-ta*, 1953, [24], 81–89; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 22,213).—The reduction at the dropping-mercury cathode of dibromoethane (**I**) and 1 : 2-dibromo-1-chloroethane (**II**) is studied; these compounds are formed by the bromination of ethylene (**III**) and vinyl chloride (**IV**) with bromine water containing 8 per cent. of methanol and saturated with NaBr. **I** and **II** are reduced polarographically, the $E_{\frac{1}{2}}$ values being $-1{\cdot}5$ V for **I** and $-0{\cdot}4$ V for **II** (*vs.* the S.C.E.); these values are independent of pH. The value of the coefficient, α, is calculated from the equation $E = E_{\frac{1}{2}} - (0{\cdot}059/\alpha)\{\log i/(i_d - i)\}$; for **I**, $\alpha = 0{\cdot}2$, and for **II**, $\alpha = 0{\cdot}34$; the reductions of **I** and **II** are therefore irreversible. The polarographic method can be used for the analysis of mixtures of **I** and **II** (and hence for **III** and **IV**). The unsaturated gaseous products are absorbed by bromine water, the excess of bromine is removed with 5 per cent. aq. NH_3 soln. and the soln. is polarographed. Gelatin (0·02 per cent.) is added to suppress the maximum. The reduction mechanisms are discussed.
E. HAYES

945. **Methylimide determination. IV. Thermal decomposition of alkyl iodide as a source of error in the micro-determination of alkyl imides.** F. Franzen, K. Eysell and H. Schall (*Mikrochim. Acta*, 1954, [6], 712–718).—The thermal decomposition of methyl iodide and ethyl iodide is a source of error in the determination of alkyl imides. If the temp. is $> 300°$ C, the effect of thermal decomposition is negligible, but at temp. $< 300°$ C non-quantitative results may arise from incomplete breakdown of quaternary ammonium compounds. The temp. of the reactants in the flask should be the same as that of the heating-bath.
A. J. MEE

946. **Determination of low hydrogen cyanide in acrylonitrile.** R. L. Maute and M. L. Owens, jun. (*Anal. Chem.*, 1954, **26** [11], 1723–1725).—Four methods for the determination of free and combined HCN, over the range of 0 to 100 p.p.m., in acrylonitrile were studied. The first method is the phenolphthalein colorimetric procedure and is recommended where an accuracy of $\pm$ 0·5 p.p.m. of total cyanide in the 0 to 10 p.p.m. range is required. The second method, Dénigès titration to a visual end-point with Ag., is the most rapid procedure and is applicable for total cyanide > 20 p.p.m. For greater accuracy and precision, especially below 20 p.p.m., the potentiometric titration with $AgNO_3$ in Na_2PO_4 buffer is satisfactory. A fourth procedure for the determination of free HCN, involving potentiometric titration in acid medium, gives precise and accurate values, without interference from HCN combined as lactonitrile.
G. P. COOK

947. **Spectrophotometric method for the estimation of amino-acids with sensitized Schiff's reagent.** S. N. Bhattacharya (*Anal. Chim. Acta*, 1954, **11** [6], 559–562).—Glycine, alanine, *nor*valine, leucine and methionine restore the colour of Schiff's reagent (Tobic's modification, *Brit. Abstr. A II*, 1942, 279). For any one of these amino-acids, the optical density of the resulting colour yields a straight line when plotted against the amount of acid present. The slope of the graph varies from one amino-acid to another and is inversely proportional to the chromatographic R_F value of the acid. The reaction is used for the spectrophotometric determination of the individual acids.
W. C. JOHNSON

948. **On the de-salting of solutions of amino-acids by ion exchange.** A. Drèze, S. Moore and E. J. Bigwood (*Anal. Chim. Acta*, 1954, **11** [6], 554–558).—Ion-exchange resins are used to separate amino-acids from sodium citrate and phosphate buffers after the amino-acids have been obtained in these media from ion-exchange chromatograms. With certain limitations, the acids can be recovered quant. from 1000 times their wt. of salts. The neutral and acidic amino-acids are absorbed on a strongly basic resin (Dowex 2) in the basic form, the resin is washed with water and the amino-acids are then eluted with *N* acetic acid. The basic amino-acids and tryptophan are absorbed on an acid resin (Dowex 50), the Na^+ are removed with 0·5 *N* HCl and the amino-acids are then eluted with 4 *N* HCl. The resulting salt-free solutions are suitable for isolation and characterisation of the amino-acids by paper chromatography.
W. C. JOHNSON

949. **The determination of thioglycollic acid in the presence of sulphite in cold-wave preparations.** F. Strache and H. J. Mierau (*Dtsch. ApothZtg.*, 1955, **95** [3], 55).—A significant loss of thioglycollic acid (**I**) occurs when an acidified soln. is boiled in an attempt to remove the sulphite commonly present in cold-wave preparations. An accuracy of $\pm$ 0·7 per cent. is claimed for the following determination of **I**. *Procedure*—To the solution (containing $\simeq$ 15 mg of **I**) in a 100-ml Erlenmeyer flask, add 70 per cent. ethanol (10 ml) acidify with 25 per cent. H_2SO_4 (1 ml) and pass CO_2 rapidly into the soln. for 30 min.; titrate with 0·1 *N* I from a microburette.
A. R. ROGERS

950. **The physicochemical determination of phenols. I. A study of the redox system Br_2 - Br'.** C. N. van Zyl and K. A. Murray (*S. Afr. Ind. Chem.*, 1954, **8** [10], 222–226).—An electrometric method for the determination of phenol in the range 12 to 0·5 p.p.m. in aqueous solution is described. The phenol is brominated with excess of bromine and potassium bromide. By measuring the redox potential of the system Br_2 - Br', the phenol concentration in a 10-ml sample can be determined with a maximum error of 13 per cent.
G. C. JONES

951. **Chromatographic separation of benzenecarboxylic acids.** R. Turriziani and S. Giovanni (*Ric. Sci.*, 1954, **24** [10], 2099–2103).—The chromatographic separation of mixtures of two or three of the following acids is described; benzoic, *iso*phthalic, phthalic and trimesic. The separation is carried out on a silica-gel column; mixtures of chloroform with 3 or 20 per cent. of *n*-butanol are used for elution. Suitable adjustment of the *p*H of the stationary phase consisting of sodium citrate - hydrochloric acid buffer and choice of the solvent make the separation of the acids possible.
S. K. LACHOWICZ

952. **Cerate oxidimetry. II. Oxidation of maleic, fumaric, salicylic and phthalic acids.** N. N. Sharma and R. C. Mehrotra (*Anal. Chim. Acta*, 1954, **11** [6], 507–511).—The method described earlier *Anal. Abstr.*, 1955, **2**, 380) is extended to the determination of maleic, fumaric, salicylic and phthalic acids. Maleic acid is heated for $\lt$ 1 hr. with an excess of 0·1 *N* $Ce(SO_4)_2$ and an equal vol. of conc. H_2SO_4; fumaric acid is treated similarly but for $\lt 2\frac{1}{2}$ hr. Benzoic acid requires 2 hr. heating, when the vol. of conc. H_2SO_4 is equal to twice the sum of the vol. of the other reactants, or $\lt$ 4 hr. heating, when an equal vol. is used. An equal vol. of conc. H_2SO_4 is used for phthalic acid and for salicylic acid and the heating period is $\lt$ 3 hr. Acetic and succinic acids are not oxidised by $Ce(SO_4)_2$, even by heating with H_2SO_4 in high concn. W. C. JOHNSON

953. **The separation and quantitative determination of ring-substituted hippuric acid derivatives.** G. L. Haberland, F. Bruns and K. I. Altman (*Biochim. Biophys. Acta*, 1954, **15** [4], 578–581).—The application of the colour reaction of hippuric acid with *p*-dimethylaminobenzaldehyde (Gaffney, Schreier, Di Ferrante and Altman, *Anal. Abstr.*, 1954, **1**, 1594) has been extended to the determination of biologically important ring-substituted hippuric acid derivatives. Hippuric acid, *o*-aminohippuric acid, *p*-aminohippuric acid and *p*-acetylaminohippuric acid were separated by paper chromatography on Whatman No. 1 filter-paper, with *n*-butanol - acetic acid - H_2O (4:1:1). After being dried in air for 1 hr., the strips were sprayed with a 4 per cent. solution of *p*-dimethylaminobenzaldehyde in acetic anhydride and again dried. Azlactone formation was completed by heating the strips at 155° C for 1·5 min. The times and temperatures are important for reproducibility. Good separation of the above compounds was achieved and the spots were eluted with three 2-ml portions of methanol. The amount of azlactone in each eluate was determined photometrically, the absorption maxima lying between 460 to 480 mμ. A mixture of *n*-pentanol - pyridine - water (35:35:30) was also used for chromatographic separation and was essential for the separation of hippuric and 2-amino-3-hydroxyaminohippuric acids. R_F values for all the compounds tested by both solvents are reported. There is a linear relation between the optical density and concentration of the azlactone up to a concentration of 50 μg in 6 ml of methanol.
G. W. CAMBRIDGE

954. **Spectrophotometric determination of aniline by means of the reaction with H-acid.** I. M. Korenman and P. A. Ganichev (*Uch. Zap. Gor'kovskogo Un-ta*, 1953, [24], 123-125; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 29,342).—To 25 ml of aniline soln. (2 to 80 μg per ml), add 1 ml of 1 *N* HCl and 5 ml of a 2 per cent. soln. of $NaNO_2$; shake and cool in ice for 30 min., shaking at 10-min. intervals. Add 6 ml of a 2 per cent. soln. of $NaHCO_3$, shake and add 0·2 ml of a 0·5 per cent. soln. of H-acid (8-amino-1-naphthol-3;6-disulphonic acid). Make up to 50 ml with water, set aside for 40 min. and measure the extinction at 600 mμ. Although the extinction does not obey Beer's law, results are satisfactory. The mean error is $\simeq$ 4 per cent. E. HAYES

955. **Spectrophotometric determination of aniline and some aromatic amino-acids.** I. M. Korenman and P. A. Ganichev (*Uch. Zap. Gor'kovskogo Un-ta*, 1953, [24], 119–122; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 29,341).—To 25 ml of an aqueous soln. of aniline (4 to 100 μg per ml), add 1 ml of *N* HCl, 5 ml of a 2 per cent. soln. of $NaNO_2$, 6 ml of a 2 per cent. soln. of $NaHCO_3$ and 4 ml of a 1 per cent. soln. of phenol; heat to 50° C for 5 min. and cool. After 10 min., measure the extinction at 470 mμ. The mean error is $\simeq$ 4 per cent. Naphthionic acid (max. 490 mμ), sulphanilic acid (max. 490 mμ) and their soluble salts can be determined in the same way. E. HAYES

956. **Correlation of ultra-violet and infra-red spectra of terpene hydrocarbons.** R. T. O'Connor and L. A. Goldblatt (*Anal. Chem.*, 1954, **26** [11], 1726–1737).—Various types of terpene hydrocarbons are readily distinguished by their u.v. spectra and can be classified into 5 groups: (*a*) saturated, (*b*) unsaturated non-conjugated, (*c*) conjugated dienes, (*d*) triene-conjugated terpene hydrocarbons, and (*e*) aromatic hydrocarbons. In general (*a*) and (*b*) compounds exhibit no characteristic absorption above 200 mμ and (*c*) compounds normally give a single max. between 255 and 290 mμ; (*d*) compounds can be distinguished from all other groups by the three sharp max. exhibited in the 260 to 280-mμ region. Group (*e*) compounds give sharp max. in the 260 to 265-mμ region; hence aromatic hydrocarbons can be distinguished from true terpenes. Ultra-violet spectra between 220 and 320 mμ of twenty-three terpene hydrocarbons are illustrated. In the "group frequency" (about 2 to 9 mμ) of the i.r., correlations of certain absorption bands show the presence or absence of certain specific groups and the absorption in the "fingerprint" region 9 to 12 mμ is characteristic of each type of molecule. Individual terpene hydrocarbons can be identified by direct comparison with standard reference compounds. G. P. COOK

957. **The determination of fluorene in tar fractions.** G. A. Vaughan and D. W. Grant (*Analyst*, 1954, **79**, 776–779).—A method is described for the determination of fluorene in coal-tar fractions. Unlike other components with the same boiling range, fluorene has a reactive methylene group enabling it to condense with certain other compounds. Of these, benzaldehyde, which condenses with fluorene in presence of an alkaline catalyst, was selected as reagent. The most suitable catalyst was found to be benzyltrimethylammonium hydroxide (Triton B). The sample is heated under reflux for 1·5 hr. with neutral benzaldehyde and absolute ethanol in the presence of Triton B. After neutralisation with 0·1 *N* HCl to bromophenol blue, hydroxylamine hydrochloride is added and the excess of benzaldehyde determined by titration with 0·1 *N* NaOH. In the place of an indicator Sb - Hg_2Cl_2 electrodes may be used to indicate the end-point if the soln. is highly coloured. From experiments with pure fluorene, a conversion of 86 per cent. should be assumed in calculations. A. O. JONES

958. **Determination of pyridine in technical 4-ethylpyridine.** István Gyenes (*Magyar Kém. Fóly.*, 1953, **59** [8], 251–252; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 31,133).—A 10-ml sample of anhydrous ethylpyridine is dissolved in 50 ml of pure benzene and the pyridine is extracted with two 50-ml quantities of water. The aqueous soln. is titrated with *N* HCl in the presence of 4 drops of dimethyl yellow and methylene blue. The volume per cent. of pyridine in the sample is given by the following empirical formula: $(v - 4{\cdot}6) \times 2{\cdot}283$, where v is the vol. in ml of *N* HCl consumed.
E. HAYES

959. **Quantitative determination of alkyl-substituted 1-methyl-4-piperidones.** A. K. Ruzhentseva and T. D. Perbacheva (*Zh. Anal. Khim., SSSR*, 1954, **9** [5], 304–307).—Alkyl-substituted 1-methyl-4-piperidones can be determined either by titration with HCl, using bromocresol green, or by oximation, after neutralisation, followed by titration of the acid set free. G. S. SMITH

960. **Unexpected results in the N-methyl [determination in] and O-methyl determination in [derivatives of] 1:2-dimethylpyridazine-3:6-dione and 2-methyl-1-phenylpyridazine-3:6-dione.** H. Gysel (*Mikrochim. Acta*, 1954, [6], 743–745).—The unreliable results obtained in the determination of N-methyl in 1:2-dimethylpyridazine-3:6-dione and 2-methyl-1-phenylpyridazine-3:6-dione are due to their partial or complete rearrangement into O-methyl compounds under the conditions of the Zeisel determination. A. J. MEE

961. **Identification of compound types in heavy petroleum gas oil.** H. E. Lumpkin and B. H. Johnson (*Anal. Chem.*, 1954, **26** [11], 1719–1722).—The major hydrocarbon and non-hydrocarbon compounds present in the aromatic portion of a heavy gas oil (600° to 1000° F) have been identified by the application of chromatographic, u.v. and mass-spectral techniques and sulphur analysis to the oil. The oil contained C_{18} to C_{35} compounds, with max. concn. in the C_{27} to C_{28} region, and the majority of sulphur compounds in an oil of high S content were found to be of the condensed aromatic-thiophen type. Illustrations showing the compound type identifications and the order of removal of the classes from an alumina-gel column are also given. G. P. COOK

962. **Chromatographic fractionation of total crude shale oil.** C. Karr, jun., W. D. Weatherford, jun., T. R. Kendrick, III and R. G. Capell (*Anal. Chem.*, 1954, **26** [11], 1841–1843).—Fractionation of crude shale oil by elution chromatography on activated alumina or bauxite adsorbents is an efficient method for recovering colourless hydrocarbon constituents free from N and containing only a little sulphur The remaining constituents can be recovered from the adsorbents as a brown-black semi-solid enriched in N and S compounds. These results indicate that the procedure could be used as a basis for crude shale oil assay or refining. G. P. COOK

963. **Determination of lanolin in powdered hand soaps.** R. Bernstein, M. Haftel and R. Grant (*Soap, N.Y.*, 1954, **30** [11], 48–49, 106).—In the specification method for determining lanolin in soap - lanolin - vegetable abrasive compositions, an aq. solution of the material is acidified and the lanolin and fatty acids are solvent extracted. Insoluble vegetable abrasive present and lanolin adsorption at the water-solvent interface make solvent extraction difficult. An improved method involves direct extraction of the lanolin with light petroleum in a Soxhlet apparatus. The recovered lanolin is then determined in the usual way by treating it with acetic anhydride - sulphuric acid and determining the colour intensity with a photo-electric colorimeter; corrections for matter extracted from the soap and vegetable abrasive, and reacting like lanolin, are made. Details of the technique are given. G. HELMS

964. **Determination of water in refrigerator oils.** R. Weber (*Kältetechnik*, 1954, **6** [10], 267–269).—The sample is shaken with anhydrous methanol; after allowing the emulsion to separate, methylene blue indicator (*cf. Brit. Abstr. C*, 1953, 194) is added and the methanol layer is titrated with Karl Fischer solution. A blank test on the methanol and a standardisation test on methanol containing 0·1 per cent. of added H_2O are carried out. A. R. PEARSON

965. **Determination of water content of refrigerator oils by Karl Fischer reagent.** H. Mandel (*Kältetechnik*, 1954, **6** [10], 269–270).—The oil sample is dissolved in $CHCl_3$ previously titrated with the reagent, in a flask with ground connections to a stirrer, burette and P_2O_5 guard tube. The precision is within 6 mg of H_2O per kg of oil, when the normal yellow-brown end-point is reached. A. R. PEARSON

966. **Detection of tri-o-tolyl phosphate and similar plasticisers in plastics.** K. Braundsorf (*Pharm. Zentralh.*, 1954, **93** [6], 222–227).—A scheme is given for the identification and rough estimation of tri-*o*-tolyl phosphate (**I**) in plastics. An ether extract of the plastic is made and tested for refractive index at 20° C, P_2O_5 content and saponification value. Qualitative tests are made after hydrolysis with ethanolic KOH, acidification and distillation to detect the presence of cresols, and with one of them (Melzer's benzaldehyde reagent) an approx. colorimetric estimation of **I** can be made by comparison with standards. Details of the methods employed are given, and results are listed for several products containing tritolyl phosphate or other plasticisers. J. H. WATON

See also Abstracts 905, 912, 918, 975, 1095.

4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

967. **Spectrophotometric micro-determination of total copper in tissues and biological fluids.** H. Jerôme and H. Schmitt (*Bull. Soc. Chim. Biol.*, 1954, **36** [9], 1343–1354).—Copper in tissues and body fluids is determined spectrophotometrically by means of the purple complex formed with 2:2′-diquinolyl in the pH range 2 to 9. The sample (*e.g.*, 1 ml of blood or plasma) is allowed to stand for 10 min. with 6 *N* HCl (0·5 ml) to liberate Cu from organic combination. Protein is pptd. by 20 per cent. trichloroacetic acid (1·5 ml) and, after 10 min., is removed by centrifuging. To 2·4 ml of the supernatant liquid are added aq. NH_3 (0·2 ml, containing $\lessapprox$ 180 g of NH_3 per litre), saturated Na acetate (1·0 ml), saturated hydroxylamine hydrochloride (0·1 ml) and ethanol (2·0 ml). After reading the density in the spectrophotometer, 0·1 per cent. ethanolic 2:2′-diquinolyl (0·1 ml) is added and the second reading made. Absorption measurements are carried out at 545 mμ ($\epsilon \simeq 6500$). In an alternative method, organic matter is destroyed with conc. H_2SO_4, HNO_3 and $HClO_4$, but slightly lower Cu values are obtained. The coloured complex, which is formed at once, is stable and can be extracted with organic solvents if required. C. E. SEARLE

968. **A new spectrographic method of determination of lead in biological materials.** M. Miki (*J. Osaka City Med. C.*, 1954, **3** [4], 370).—A small amount of lead in org. materials (especially in blood) can be determined with the aid of the interrupted-arc method. *Procedure*—The sample ($\simeq$ 1 ml of

blood) is dried and ignited at a temp. below 500° C in a silica crucible. A few drops of 0·1 per cent. of HNO_3 are added to the crucible and lead is dissolved by gentle heating. An interrupted-arc is put between carbon electrodes impregnated with 0·02 g of 0·1 per cent. HNO_3. (Current, 5 amp.; interruption factor, 1/4; interruption period, 3 sec.; internal standard, bismuthyl nitrate; exposure, 7 min.; line pair, Pb 2833 ; Bi 2898.) The results compared favourably with values obtained by the condensed-spark method and by the continuous-arc method. K. SAITO

969. **Estimation of very small amounts of iron in biological material with o-phenanthroline hydrochloride in the presence of tartaric acid.** H. Kathen (*Biochem. Z.*, 1954, **325** [6], 491–496).—Observations on possible sources of error in the estimation of serum iron by the *o*-phenanthroline method (Heilmeyer and Plötner, "Das Serum-Eisen und die Eisenmangelkrankeit," Fischer, 1937) are reported. The interference of soluble iron complexes with the quantitative reaction must be considered. Wet ashing produced marked loss of Fe; this could be diminished but not abolished by addition of tartaric acid, but dry ashing for 4 hours at 700° C appears to be the most suitable method; the presence of tartaric acid eliminates error in this method also. G. W. CAMBRIDGE

970. **Determination of ammonia in the cold in media rich in urea. Application to urine.** M. Rangier and M. Krebs (*Bull. Soc. Chim. Biol.*, 1954, **36** [10], 1479–1485).—A method is described for determination of ammonia in urine, based on pptn. of the iodomercuric complex. The reagent consists of an aq. solution of $HgCl_2$, KI, KCl and KOH, which precipitates the NH_3 completely but has no effect on the urea. The ppt. is washed, dissolved in aq. KI, rendered slightly acid with HCl (to eliminate Hg formed by the action of reducing substances in the urine) and the Hg is determined as HgS after pptn. with $(NH_4)_2S$. Each mg of HgS corresponds to 0·0325 mg of NH_3. The NH_3 can also be determined by dissolving the complex in dil. HCl (containing no KI), the solution being made alkaline and then treated with the $HgCl_2$ - KI - KCl reagent. The turbidity of the solution is examined photo-electrically and compared with that given by known amounts of NH_3. J. N. ASHLEY

971. **Determination of ammonia in the cold in media rich in urea. Application to blood plasma.** M. Rangier and G. Thiroux (*Bull. Soc. Chim. Biol.*, 1954, **36** [10], 1487–1490).—The method depends on pptn. of the NH_3 as the iodomercuric complex. Blood is collected in the presence of heparin and deproteinisation is effected with Na tungstate - H_2SO_4. The liquid is then treated with aq. $HgCl_2$ - KI - KCl - KOH, and the NH_3 complex is separated by centrifuging; it is dissolved in dil. HCl and the mixture is centrifuged to eliminate reduced Hg. Since the amount of NH_3 present is very small, it is determined turbidimetrically by the method suggested for urine (see Abstract 970). J. N. ASHLEY

972. **Colorimetric micro-determination of methanol in the atmosphere and biological media. Simultaneous determination of formaldehyde and formic acid.** R. Fabre, R. Truhaut and A. Singerman (*Ann. Pharm. Franç.*, 1954, **12** [6], 409–428).—The possible methods for the estimation of methanol, formaldehyde and formic acid, with particular reference to the investigation of methanol intoxication, are discussed. A method is described in which chromotropic acid is used. At 0° C, 3 ml of a solution containing 3 to 18 μg of formaldehyde is treated with 5 ml of 5 per cent. chromotropic acid. The solution is then heated on a boiling water-bath for 30 min., cooled, made up to 10 ml with 9 *M* H_2SO_4 and the extinction is measured with a yellow (Pulfrich S 57) filter. For methanol, 2 ml of the solution (containing 10 to 60 μg) are oxidised at 0° C for 5 min. with 0·05 ml of 5 per cent. $KMnO_4$ in 0·5 ml of 85 per cent. H_3PO_4. Excess of $KMnO_4$ is removed with $NaHSO_3$ (avoiding excess), and the formaldehyde produced is estimated as before. For formic acid, 80 mg of Mg ribbon and 0·5 ml of conc. HCl (in 0·05-ml portions at 1 min. intervals) are added to 2 ml of soln. (containing 10 to 60 μg); one minute after the last addition of HCl, the formaldehyde is estimated as before. Typical calibration curves are shown. The application of the determination to industrial atmospheres and to biological fluids is discussed. Only methanol can be recovered quantitatively from the latter; a method is given for distillation of methanol or its entrainment in a current of air from preparations of tissue, blood or urine treated with picric acid. E. J. H. BIRCH

973. **Paper chromatography of the non-volatile organic acids in biological fluids. I. Urine. Chromatographic technique.** R. Nordmann, O. Gauchery, J. du Ruisseau, Y. Thomas and J. Nordmann (*Bull. Soc. Chim. Biol.*, 1954, **36** [10], 1461–1471).—A method is described for the detection of non-volatile org. acids in urine. Interfering substances, *e.g.*, cations, ampholytes, non-ionic substances and some inorg. anions, are removed by passing the urine through a column of Dowex 2. This is better than electrolytic de-salting, which causes loss, whilst extraction of the org. acids with ether is incomplete. The acids are eluted from the column with 6 *N* formic acid, and after concentration of the eluate, an aliquot equivalent to an amount of urine containing 2 μg of creatinine is transferred to paper and is chromatographed in two dimensions by use of first a basic solvent (ethanol - aq. NH_3) and then an acidic solvent (propanol - formic acid - cineole-water). The spots due to the various acids are detected by observation in u.v. light, spraying with various pH indicators or by determination of R_F. J. N. ASHLEY

974. **A rapid estimation method of urinary ethereal sulphate by a phototurbidimeter.** T. Fujii and C. Nakagawa (*J. Osaka City Med. C.*, 1954, **3** [2], 189).—Sperber's turbidimetric method for the estimation of ethereal sulphate (*J. Biol. Chem.*, 1921, **47**, 56) was slightly modified for use with a phototurbidimeter. The transmission of a $BaSO_4$ suspension remains constant from 6 to 20 min. after the addition of $BaCl_2$; it is proportional to the concn. of SO_4'' within the range of 4 to 30 μg of SO_3 per ml. This method decreases the time required for an estimation. K. SAITO

975. **Iodimetric determination of uric acid.** W. D. Treadwell and A. Wahl (*Helv. Chim. Acta*, 1954 [7], **37**, 1948–1954).—The iodimetric titration of uric acid in saturated $NaHCO_3$ solution in the presence of starch gives satisfactory results, 1 mol. of acid being oxidised by 1 mol. of I. For the purpose of comparison, the titration in acid solution with bromide - bromate and Folin's colorimetric determination with 18-tungsto-2-phosphoric acid in KCN solution are used. Preliminary experiments point to the possibility of using an amperometric determination for small concn. of uric acid, a polished platinum

electrode functioning as anode. At $\simeq 80°$ C, alloxan is readily reduced quantitatively to dialuric acid by a Cd filter. Iodimetric oxidation of this acid in HCO_3' solution reforms alloxan. In contrast to free dialuric acid, the iodimetric oxidation of its mol. compound with alloxan (alloxanthin) to alloxan occurs in two sharply defined univalent stages. H. WREN

976. **The estimation of Darstine [1-(3-hydroxy-5-methyl-4-phenylhexyl)-1-methylpiperidinium bromide] and its tertiary analogue, 5-methyl-4-phenyl-1-(1-piperidyl)-3-hexanol hydrochloride, in plasma and urine.** E. K. Tillson, S. F. Paulson and K. H. Beyer (*J. Lab. Clin. Med.*, 1954, **44** [6], 880–884).—A macro method for the colorimetric determination of mepiperphenidol (Darstine) (**I**) and its corresponding piperidine tertiary analogue (**II**) is based on the formation of a coloured complex with methyl orange. The complex is extracted with ethylene dichloride, acidified and estimated colorimetrically. Calibration is rectilinear up to 6 mg per 100 ml for both **I** and **II**, but for **I** in plasma it is about 10 per cent. lower than in aqueous solutions. A test for differentiating between **I** and **II** and a micro adaptation of the procedure suitable for **II** only are also described. W. H. C. SHAW

977. **A new method for analysis of plasma fibrinogen, utilising a cationic detergent.** R. F. Jacox (*J. Lab. Clin. Med.*, 1954, **44** [6], 885–889).—In the method described, the fibrinogen pptd. from 1 ml of oxalated plasma with aq. ammonium sulphate is separated by centrifuging and then redissolved in 1 per cent. aq. NaCl. An aliquot of this soln. is treated with 0·1 *M* phosphate buffer (pH 5·1) containing 0·4 *M* NaCl, and aq. 0·1 per cent. benzyldimethyloctadecylammonium chloride is added. The turbidity formed in 10 to 15 min. is estimated photo-electrically to give an apparent optical density that is proportional to fibrinogen concentration. The method gives 103·7 (± 6·1) per cent. recovery of fibrinogen added to abnormal sera in concentrations between 90 and 900 mg per 100 ml, but results are generally 10 to 30 per cent. higher than those given by a conventional clotting technique. The reasons for the discrepancies are discussed. W. H. C. SHAW

978. **Separation of oestrogens in urinary extracts by partition chromatography.** W. S. Bauld (*Biochem. J.*, 1955, **59** [2], 294–300).—A method is described for the separation and purification of oestriol, oestrone and oestra-3:17β-diol in extracts of hydrolysed urine. The oestriol is removed from a benzene extract by treatment with water, and is then separated from hydrophilic contaminants on a partition chromatogram by using Celite 535 and methanol - ethylene dichloride (70:30, by vol.). The oestrone and the diol are then separated by partition chromatography, with *N* NaOH as the stationary phase and benzene as the mobile phase. Elution of the diol is best achieved with a mixture of ethylene dichloride and benzene (3:1 by vol.). The main factors that affect the elution of oestrogens from partition chromatograms are described fully. J. N. ASHLEY

979. **A method for spectrophotometric determination of chenodeoxycholic acid in bile.** B. Isaksson (*Acta Chem. Scand.*, 1954, **8** [6], 889–897).—Chenodeoxycholic acid (**I**) dissolved in pure ethyl acetate gives, with the Liebermann - Burchard reaction, a colour having an absorption peak at 610 to 620 mμ; the presence of other bile acids has no influence and the absorbancy obeys the Beer - Lambert law. *Procedure*—Hydrolyse about 0·2 to 1 ml of fresh bile with NaOH, acidify with HCl and extract the bile acids with ether. Evaporate the ether, dissolve the residue in absolute ethanol and make up to 10 ml. Evaporate an aliquot of the soln. in a test tube and prepare a series of standards containing 0·2 to 1·5 mg of **I** in similar tubes. Dry at 100° C for 5 min., cool and add to each tube 3 ml of ethyl acetate - H_2SO_4 (15:1 by vol.). Cool and add 2 ml of acetic anhydride, mix and, 12 to 15 min. after the addition, determine the absorbancy at 615 mμ. Hence calculate the **I** in the bile. A micro method is also described. Duplicate determinations on 26 extracts gave a standard error of ± 2·4 per cent. N. E.

980. **Tissue chloride.** W. D. Shenk (*Arch. Biochem. Biophys.*, 1954, **49** [1], 138–148).—The van Slyke method (*J. Biol. Chem.*, 1923, **58**, 523) for the determination of chloride, although giving satisfactory results for blood, is unsatisfactory for tissues. The low results may be due to the loss of Cl as chloranil ($C_6Cl_4O_2$) or to incomplete digestion of the tissue. Much higher values are obtained with the Parr bomb or the closed Carius-micro method. N. E.

981. **The quantitative estimation of reducing sugars on paper chromatograms.** F. G. Fisher and H. Dörfel (*Hoppe-Seyl. Z.*, 1954, **297** [3–6], 164).—The colour developed on heating reducing sugars with triphenyltetrazolium bromide (**I**) in alkaline soln. is used for their estimation. The reaction is not stoicheiometric and results depend largely on the temp. of development. The paper blank is small only under fixed conditions. There is proportionality between sugar concn. and colour formation only when the reagent is present in great excess. Before the chromatography, it is preferable to determine the total sugar and the total aldose content. Each single component of the mixture applied to the paper should be in concn. of 0·2 to 0·5 per cent.; when there are great differences in concn. of different monosaccharides, several dilutions may be used. Known monosaccharides in 2 per cent. soln. are run as standards. Ethyl acetate - pyridine - water (40:11:6, by vol.) is used as developing solvent. Descending chromatograms are run for 20 hr. at 20° C and dried in an air stream at room temp. The paper is soaked (not sprayed) in a mixture of equal parts of *N* methanolic NaOH and 4 per cent. **I** in methanol. It is then dried in a current of air at room temp. and the colour developed by heating for 60 min. at 65° C or 30 min. at 70° C in an insulated double-walled copper chamber whose construction is described. The formazan spots are cut out immediately after development and eluted with a mixture of 10 vol. of methanol and 1 vol. of glacial acetic acid. The extinction is measured at 485 mμ against the eluent as blank; a graphical method of determining the blank value to be subtracted for the paper is described. F. POWELL

982. **Colorimetric micro-determination of ammoniacal nitrogen.** B. Lubochinsky and J.-P. Zalta (*Bull. Soc. Chim. Biol.*, 1954, **36** [9], 1363–1366).—Ammonia (0·5 to 10 μg), resulting from the Kjeldahl destruction of organic material containing N, is determined spectrophotometrically by means of the stable blue colour formed by reaction with phenol and hypochlorite in the presence of nitroprusside as catalyst. The sample, in a 20-ml calibrated flask, is just neutralised to methyl red with very

dil. NaOH. To the soln. are added 5 per cent. Na phenoxide (2 ml), 0·1 *M* phosphate buffer (pH 12) (2 ml), freshly prepared 0·5 per cent. sodium nitroprusside soln. (2 ml) and NaOCl with 4 per cent. of available Cl (0·5 ml). The soln. is made up to 20 ml, and the colour is allowed to develop in the dark for 30 min. The density is measured at 610 mμ, or alternatively a colorimeter with a red filter may be used. Results are reproducible to within 2 per cent.
C. E. SEARLE

983. **Some remarks on the paper chromatography of amino-acids. (Preliminary communication.)** M. T. Beck and P. Ebrey (*Acta Chim. Acad. Sci. Hung.*, 1954, **4** [2–4], 231–233).—The statement of Zimmermann (*Z. anal. Chem.*, 1953, **138**, 321) that amino-acids exert an influence on the R_F values of each other is investigated. Evaluation of mixtures of glycine and glutamic acid based on the R_F values and the area of the spot gave inconsistent quant. and qual. results. Similar observations were made with other pairs of amino-acids. The results are attributed to interaction between the amino-acids, because with certain proportions of glycine and glutamic acid a new spot was seen, corresponding in position to serine.
N. E.

984. **Chromatography of amino-acids on ion-exchange resins. Use of volatile acids for elution.** C. H. W. Hirs, S. Moore and W. H. Stein (*J. Amer. Chem. Soc.*, 1954, **76** [23], 6063–6065).—Pure amino-acids from protein hydrolysates are separated by a two-stage chromatographic procedure. Aspartic acid, glutamic acid and tyrosine are first separated on a 30-cm column of the acetate form of the strongly basic Dowex 1-X8 resin, with 0·5 *N* acetic acid at 25° C as the eluent. The remaining amino-acids are separated on a 150-cm Dowex 50-X4 resin column by elution with *N* HCl at 25° C until the glycine has emerged, and then with HCl increasing from *N* to 4 *N* at 50° C. The recoveries of glutamic and aspartic acids from the Dowex 1-X8 column are only 85 per cent. and 70 per cent., respectively. The reason for the loss is not known, and none is carried through to the second stage. The recoveries from the Dowex 50-X4 are quant., except for methionine, when a portion may undergo oxidation to sulphoxide during the hydrolysis and chromatography.
J. H. WATON

985. **Procedures for the chromatographic determination of amino-acids on four per cent. cross-linked sulphonated polystyrene resins.** S. Moore and W. H. Stein (*J. Biol. Chem.*, 1954, **211** [2], 893–906).—A method is described for chromatographic separation of ninhydrin-positive compounds on 150-cm × 0·9-cm columns of 4 per cent. cross-linked sulphonated polystyrene (Dowex 50-X4). It permits recovery of most amino-acids within 3 per cent. of the theoretical recovery in a single chromatogram at pH 3·1 to 5·1, by elution with Na acetate - citrate buffers of gradually increasing pH and ionic strength. The concn. of amino-acids in the effluent fractions are determined photometrically by a modified ninhydrin reagent. Use of an eluent of continuously changing composition eliminates loss of resolving power, which occurs immediately after stepwise changes in the composition of the eluent; the chromatograms are also more convenient to operate. Use of a resin of lower cross linking than that of Dowex 50-X8 makes the column applicable to the separation of peptides as well as of amino-acids. The increased resolving power of this method renders the procedure very useful for studies on the amino-acids and related compounds of blood plasma and tissue extracts, as well as for the determination of the amino-acid composition of proteins.
J. N. ASHLEY

986. **A modified ninhydrin reagent for the photometric determination of amino-acids and related compounds.** S. Moore and W. H. Stein (*J. Biol. Chem.*, 1954, **211** [2], 907–913).—A modified ninhydrin reagent is described for the determination, by the photometric method, of amino-acids and related compounds. It consists of 2 per cent. of ninhydrin and 0·3 per cent. of hydrindantin in 2-methoxyethanol - 4 *N* Na acetate buffer (pH 5·5) (3:1). The strength of the buffer eliminates or decreases the need for preliminary adjustment of the pH of samples during analysis of effluent fractions obtained in ion-exchange chromatography. The modified reagent also eliminates the need to use $SnCl_2$ (because of the direct addition of hydrindantin) and thus avoids the pptn. of Sn salts, which occurs when the earlier reagent is used for the analysis of samples of the PO_4''' buffers frequently employed in the chromatography of proteins. The colour yields from amino-acids and related compounds are similar in most cases to those obtained with the earlier reagent.
J. N. ASHLEY

987. **The estimation of carnitine.** E. Strack and I. Lorentz (*Hoppe-Seyl. Z.*, 1954, **298** [1–2], 27).—Paper chromatography with ethanol - aq. NH_3 - H_2O mixtures can be used to separate L-carnitine from the similar naturally occurring bases glycylbetaine and choline. Paper electrophoresis in citrate - HCl buffer at pH 3·0 to 3·8 may also be used. The position of the carnitine is detected by soaking the paper in 0·5 per cent. ethereal iodine soln. and cutting out the spots. Heating with conc. H_2SO_4 converts carnitine to crotonic acid betaine, which can be estimated by permanganate titration or by the biological activity of its methyl ester.
F. POWELL

988. **Studies in histochemistry. XXXI. A method for the determination of protein in millimicrogram quantities.** S. N. Nayyar and D. Glick (*J. Histochem. Cytochem.*, 1954, **2** [4], 282–290).—The method of Greif (*Proc. Soc. Exp. Biol. Med.*, 1950, **75**, 813) has been modified; protein is quant. pptd. with an excess of Bromsulphthalein at a pH of 2·5 and the excess of dye is determined photometrically at 580 mμ. A standard deviation of ± 5·3 per cent., corresponding to 3·4 mμg of protein nitrogen, was observed.
N. E.

989. **The determination of nucleic acids in epithelial tissue.** J. Hirtz and M. T. Fayet (*Bull. Soc. Chim. Biol.*, 1954, **36** [10], 1447–1451).—The sources of error in the method of Schneider (*Brit. Abstr. C*, 1946, 120) are studied; the main causes are degradation of the nucleic acids during grinding of the tissue and the difficulty of extracting the acids completely from the disintegrated tissue. Modifications of the method are used with epithelial tissue, *e.g.*, from ox tongue. The tissue, which is cut into very small pieces, is put into 95 per cent. ethanol at −25° C, and the mixture is ground with quartz in a mortar cooled to −25° C. The resulting mixture is centrifuged and the acids are extracted from the solid residue. Two extractions with cold 5 per cent. aqueous trichloroacetic acid give the acid sol. fraction which contains the ribonucleic acid, and then extraction with 5 per cent. aq. trichloroacetic acid at 90° C gives the deoxyribonucleic acid. Both acids are then determined by standard methods.
J. N. ASHLEY

990. **The preparation of apurinic acid and a new method for the quantitative determination of the purine contents of deoxyribonucleic acids.** S. G. Laland (*Acta Chem. Scand.*, 1954, **8** [3], 449–458).—The contact of an aq. soln. of deoxyribonucleic acid (DNA) with Amberlite IR-120(H) results in the liberation of purines (retained by the resin) and apurinic acid (found in the filtrate). DNA (approx. 5 mg) is dissolved in H_2O (5 ml) and Amberlite IR-120(H) (0·72 g) is added. The flask is closed and shaken for 12 days at room temp.; the resin is transferred with water (20 ml) to a small glass column fitted with a glass disc, and washed with water (20 ml); it is then eluted with 4 *N* HCl at a rate of 1 ml per min., the vol. being made up to 100 ml. The u.v. absorption of the eluate is determined at 270 and 250 mμ, and by means of a formula, the adenine and guanine are calculated. N. E.

991. **The estimation of DPN/DPNH ratio in living cells and tissues.** H. Holzer, S. Goldschmidt, W. Lamfrecht and E. Helmreich (*Hoppe-Seyl. Z.*, 1954, **297** [1], 1–18).—The reduced form of diphosphopyridine nucleotide, DPNH (**I**), has a strong band at 340 mμ that the unreduced form, DPN (**II**), does not possess. This together with the different stabilities in acid and alkali and the alcohol-dehydrogenase (**III**) catalysed oxidation of ethanol in presence of **II** to give acetaldehyde and **I** forms the basis of a method that is claimed to be specific, distinguishing **II** from triphosphopyridine nucleotide (**IV**), as **III** does not react with **IV**. Details are given for liver and yeast. *Procedure for* **II** *in liver*—A portion (about 2 g) is weighed and homogenised with trichloroacetic acid (5 per cent. in the proportion of 1 g to 5 ml). After setting it aside for 5 min. at room temp., centrifuge the homogenate at 2° to 5° C for 5 to 10 min. at 15,000 r.p.m., then neutralise with twice its vol. of 0·25 *N* Na_3PO_4, mix and adjust the pH to 6·2 to 6·4. Take 3 ml of centrifugate and add 0·6 ml of a mixture of equal vol. 0·1 *M* aq. semicarbazide hydrochloride and 96 per cent. ethanol. Then add 0·002 ml of a suspension of **III** in 60 per cent. saturated aq. ammonium sulphate. Measure at 340 mμ or 366 mμ. Then μg per g of liver are equivalent to $E_{340} \times 1{\cdot}67$ or $E_{366} \times 2{\cdot}96$. *Procedure for* **I** *in liver*—Homogenise another piece of liver with *N* NaOH at 100° C (4 ml per g of liver). Neutralise with 2·8 ml of *M* KH_2PO_4 per ml and adjust the pH to 6·2 to 6·4. Centrifuge as above. Take 3·8 ml, add 0·1 ml of *M* acetaldehyde and 0·002 ml of **III**. Then μg per g of liver $\equiv E_{340} \times 2{\cdot}04$ or $E_{366} \times 3{\cdot}61$. Procedures for yeast are also given. F. Powell

992. **The estimation of thiols and disulphides by potentiometric titration with silver nitrate.** R. Cecil and J. R. McPhee (*Biochem. J.*, 1955, **59** [2], 234–240).—Titrations of thiols with $AgNO_3$ are carried out in buffered solutions at two ranges of pH. One group of thiols is titrated at pH 1·8 to 2·2 by using a silver electrode; another group is titrated at pH 6·5 to 7·0 by using the appropriate silver thiol electrode. The buffers (0·01 to 0·02 *M*) are H_2SO_4 or HNO_3 in the acid range and phosphate in the neutral range. The preparation of the silver thiol electrodes is described and a simple thermodynamic treatment is given. The method is applied to the determination of six disulphides. They are first reduced with Na_2SO_3 to give one equivalent of the corresponding thiol. The factors that govern the optimum pH for this reaction are discussed, and the conditions for each disulphide are given. This work discloses errors in previous work on the determination of thiols and disulphides. These are discussed and corrections are given. J. N. Ashley

993. **Acetylated Sudan black B as a reagent for lipids.** W. G. B. Casselman (*Biochim. Biophys. Acta*, 1954, **14** [3], 450).—Acetylated Sudan black B (**I**) is useful for locating lipids in spot tests or paper chromatograms; it is more specific than the parent dye. Acetylation is effected with an equivalent of acetic anhydride in ether solution. **I** may be applied as a saturated solution in 70 per cent. ethanol or preferably in propylene glycol. N. E.

994. **The micro-estimation of neutral 17-ketosteroids.** W. Zimmermann and D. Pontius (*Hoppe-Seyl. Z.*, 1954, **297** [3–6], 157).—Tables are given summarising the details of the procedure and the working time required in seven different micromethods including the one presented. Diagrams are given of simply constructed devices for carrying out the extraction and evaporation stages of the method. *Procedure*—The urine (10 ml) is heated to boiling for 20 min. in a glycerol bath in a stoppered tube 3 cm × 18 cm with conc. HCl (1 ml) and 10 per cent. aq. $CuSO_4 . 5H_2O$ (1 ml). After being cooled, the liquid is extracted with 20 ml of ether and then the aq. layer is removed by suction. The ether is washed with 20 ml of 10 per cent. w/v NaOH soln., twice with water and dried over sodium sulphate. The evaporated extract is dissolved in ethanol (2 ml) and the colour developed with 2 per cent. w/v ethanolic *m*-dinitrobenzene and 3 *N* aq. KOH for 90 min. at 25° C. The ketosteroid colour is extracted with ether before measuring the extinction at 520 mμ. F. Powell

995. **On the microbiological evaluation of the enzyme - substrate system alliin - alliinase.** P. Klein and C. Souverein (*Biochem. Z.*, 1954, **326** [2], 123–131).—Alliin is a sulphur-containing compound extracted from *Allium sativum* L. (Cavallito, Buck and Suter, *J. Amer. Chem. Soc.*, 1944, **66**, 1952). It is broken down by alliinase to allicin, pyruvic acid and ammonia. The inhibitory action of allicin on the growth of *B. subtilis* has been employed as a basis for bio-assay and definition of alliinase activity on the basis of such tests is reported. G. W. Cambridge

996. **Application of chromatography. XX. Quantitative determination of cocarboxylase preparations.** T. Masuda and M. Asai (*Pharm. Bull., Japan*, 1954, **2** [3], 287–291).—The purity of cocarboxylase prep. was determined by paper electrophoresis at pH 3·0 in Theorell buffer. The bands of cocarboxylase (TDP) and the monophosphate (TMP) were detected by u.v. light, cut out and analysed for N and P after extracting with acetic buffer pH 4 at 100° C for 30 min. Thiamine was determined directly in aq. solution by the cyanogen bromide method. H. F. W. Kirkpatrick

997. **Studies in histochemistry. XXXVI. Determination of millimicrogram quantities of co-enzyme A and its distribution in the rat adrenal.** B. G. Malmström and D. Glick (*J. Biol. Chem.*, 1954, **211** [2], 677–686).—A histochemical method is described for the determination of μg amounts of co-enzyme A in tissues. It is based on the method of Kaplan *et al.* (*Brit. Abstr. A III*, 1948, 1091). Conditions needed for measurements on the microscale are elaborated. A new more advantageous coenzyme-A unit is suggested, which equals 1·4 Kaplan - Lipmann units and is equivalent to $\simeq 1$ μg of bound

pantothenic acid. By decreasing the reaction vol., a 50-fold increase in sensitivity is attained; the sensitivity of the micro-method is approx. 3 milliunits or 10 mμg of co-enzyme A. J. N. ASHLEY

998. **Quantitative determination of catalytic activity of biological material.** H. Lück (*Enzymologia*, 1954, **17** [1], 31-40).—Difficulties in the determination of the catalytic activity of biological material are described and methods of obtaining the most reliable results are given. Storage and extraction time are important. For accurate results, the enzyme must be in a homogeneous soln. The plant or animal tissue should be sectioned in a freezing microtome to 20 to 50 μ so that nearly every cell is injured, instead of grinding the material in a mortar with sand or glass. Increased activity of the enzyme may occur by destroying cells of unicellular organisms. The use of the velocity constant of a monomolecular reaction, the max. amount of H_2O_2 destroyed by the catalase and the max. reaction velocity as a measure of activity are considered. The substrate concn. at zero time is found by extrapolation, when the monomolecular velocity constant, is used. The proportionality of the activity constant to the amount of enzyme present in the biological material is discussed. P. HAAS

999. **The absorption spectra of suspensions of living micro-organisms.** K. Shibata, A. A. Benson and M. Calvin (*Biochim. Biophys. Acta*, 1954, **15** [4], 461–470).—The "total absorbance" of a suspension can be measured by a simple modification of a spectrophotometer without any change to the optical system or treatment of the suspension. It is achieved by the attachment of opalescent plates to the cuvettes for both solvent and suspension. Clear absorption spectra of micro-organisms, blood and translucent materials can be obtained by this technique. G. W. CAMBRIDGE

See also Abstracts 854, 948, 953, 1075, 1077.

Drugs

1000. **Determination of ash in vegetable drugs.** G. Bernsen (*Dansk Tidsskr. Farm.*, 1954, **28** [11], 245–260).—Conditions for the expeditious and accurate determination of total and acid-sol. ash by the use of simple equipment are critically examined. The sample (≃ 2 g) is incinerated in a flat 9-cm silica dish over an Argand burner, the max. temp. being kept below dull red heat. The dish should be cooled in a desiccator during < 1 hr., and weighing should be accomplished rapidly. For the determination of acid-sol. ash, it is sufficient to heat the ash in the dish to the b.p. with two successive portions of 25 ml of 2 *N* HCl; the filter-paper with the insol. ash is incinerated in the dish, and (to eliminate possible error due to flaking of the dish) the original wt. of the dish is used in the calculation. The insol. ash should only be collected in a Gooch crucible when a platinum dish is used. P. S. ARUP

1001. **Mercurimetric analysis of some pharmaceutical preparations.** L. A. Tetyueva (*Aptechnoe Delo*, 1954, **3** [5], 12–14).—Many halogen-containing pharmaceutical preparations can be determined by titration with $Hg(NO_3)_2$; Na nitroprusside, diphenylcarbazide or 2-nitroso-1-naphthol is used as indicator. The mercurimetric method gives results agreeing with the argentimetric method for the following compounds: NaCl, $CaCl_2$, KBr, carbachol, procaine hydrochloride and (after destruction of organic matter) bromvaletone, tribromoethanol and acriflavine hydrochloride. For the assay of amyl nitrite, 10 ml of ≃ 0·1 *N* amyl nitrite are added to a mixture of 10 ml of 5 per cent. $KClO_3$ soln. and 5 ml of dilute HNO_3; the mixture is set aside for 30 min. and titrated with 0·1 *N* $Hg(NO_3)_2$, 3 drops of 10 per cent. aqueous sodium nitroprusside soln. being added as indicator. E. HAYES

1002. **Applicability of titration in non-aqueous solvents to pharmaceuticals.** E. G. Wollish, C. W. Pifer and M. Schmall (*Anal. Chem.*, 1954, **26** [11], 1704–1705).—A short survey of the application of titration in non-aqueous solvents for the determination of pharmaceuticals is given. The effects of interferences by other compounds in various pharmaceutical preparations are discussed, and means to overcome them are suggested. A technique is described for the determination of optical rotation, when the angle to be read is of low magnitude, the quantity to be determined is small or the concentration of the salt in water is limited by its low solubility. G. P. COOK

1003. **Tables for the identification of N.F. 10 crystalline substances by the microscopic-crystallographic method.** A. H. Tillson and W. V. Eisenberg (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [12], 760–767).—Optical crystallographic data are given for 184 substances in the [United States] National Formulary 10. A. R. ROGERS

1004. **A note on the detection of atropine sulphate in the presence of butacaine sulphate.** W. E. O'Malley, J. W. Forrest and J. C. Krantz, jun. (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [12], 769).—Atropine may be detected in the presence of butacaine by warming the mixture with conc. H_2SO_4. Phenylethanol (odour of roses) is evolved; the odour changes to that of benzaldehyde on the addition of $K_2Cr_2O_7$. A. R. ROGERS

1005. **Ultra-violet spectrophotometric determination of reserpine.** E. H. Sakal and E. J. Merrill (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [12], 709–715).—Reserpine may be determined by measurement of its u.v. absorption at 267 mμ in 5 *N* acetic acid; $E^{1\%}_{1\,cm}$ at 267 mμ = 278. The effect of linear irrelevant absorption may be corrected by the equation:

$$E_{267}\ (\text{corr.}) = 7{\cdot}802\ E_{267} - 3{\cdot}840\ E_{275} - 3{\cdot}963\ E_{259{\cdot}25}$$

Crude extracts of rauwolfia root require preliminary purification by paper electrophoresis in 5 *N* acetic acid followed by elution of the reserpine zone (made visible by fluorescence under u.v. light) with 5 *N* acetic acid. In all samples examined, the reserpine was the fluorescent zone of least mobility, and the absorption spectrum of irrelevant matter eluted simultaneously was substantially linear over the range 259·25 to 275 mμ. The reproducibility of the paper-electrophoretic assay is ≃ ± 2·5 per cent. Reserpine tablets may be assayed by extraction with methanol and measurement of u.v. absorption; with methanol as solvent, $E^{1\%}_{1\,cm}$ at 267 mμ = 278, and $E_{267}\ (\text{corr.}) = 8{\cdot}093\ E_{267} - 3{\cdot}983\ E_{275} - 4{\cdot}111\ E_{259{\cdot}25}$. The methanol extract of tablets containing mixed rauwolfia alkaloids should be purified by paper electrophoresis before spectrophotometric assay. A. R. ROGERS

1006. **The assay of ergot and its preparations.** G. E. Foster (*J. Pharm. Pharmacol.*, 1955, **7** [1], 1–15).—A review of chemical, physico-chemical and

biological methods for the assay of ergot and its extracts and preparations of the ergot alkaloids is given. Analytical applications in commerce are also discussed. (63 references.) A. R. ROGERS

1007. **Paper chromatography of digitalis glycosides.** Zengo Kanda and Atsushi Sekiya (*Jap. J. Pharmacol.*, 1954, **4** [1], 10–11).—Two commercial preparations of digitoxin were treated by chromatography on silica gel, and the fractions giving a positive Keller - Kiliani reaction were tested. Both showed the absence of gitoxin on the paper chromatogram, and both gave positive tests with the Baljet, Legal and Raymond reagents, but only one of them gave a fluorescent spot on the paper chromatogram with the Svendsen - Jensen reagent (*Pharm. Acta Helv.*, 1950, **25**, 241) (chloroform - methanol - trichloroacetic acid). The last-named reagent should not be relied upon without confirmation by use of other reagents. The purified specimens had similar LD_{50} on frog heart.
S. C. I. ABSTR.

1008. **Comparative study of the determination of cantharidin in cantharides.** C. G. van Arkel and M. Meyst (*Pharm. Weekbl.*, 1955, **90** [2], 38–42).—The following methods for determining cantharidin in cantharides are compared: the method given in the Dutch Pharmacopoeia V, the methods of du Bois, of Fischer and Ruess, of Hecht and Parks, of Schoorl, and that described in the Danish Pharmacopœia IX (Addendum Veterinarium). The method given in the Danish Pharmacopoeia is recommended. *Procedure*—A sample of 15 g of powdered cantharides is allowed to stand with 148·5 g of chloroform and 2 ml of HCl (25 per cent.) for 16 to 20 hr., and the mixture is then shaken for 2 to 3 hr. A 10-g portion of the chloroform extract is filtered into a weighed flask, and the solvent is removed by distillation and finally in a current of warm air. The residue is boiled with 100 ml of H_2O for 5 min. under reflux, and filtered through a wet paper. The paper is returned to the flask and the boiling repeated with 50 ml of H_2O. The liquid is filtered through a wet filter, and the paper and flask are washed with 50 ml of H_2O. The solution is acidified with 2 ml of HCl and shaken with three 25-ml portions of chloroform. The solvent is removed as before, and 5 ml of a mixture of 9·5 ml of light petroleum (boiling range 40° to 60° C) and 0·5 ml of ethanol are added. After standing for half an hour, the liquid is decanted through cotton-wool, and the washing is repeated twice with the petroleum mixture, the mixture being set aside for 10 min. each time. The cotton wool is washed with 4 ml of chloroform and the solvent is removed. The crystals are dried for 30 min. at 60° C and weighed. A correction of 10 mg is added to the weight.
A. J. MEE

1009. **A new method of large-scale preparation of hypertensin, with a note on its assay.** W. S. Peart (*Biochem. J.*, 1955, **59** [2], 300–302).—A reliable sensitive method is described for the biological assay of hypertensin by the use of the pressor responses in the anaesthetised rat. The method is sensitive to small amounts of hypertensin and gives reproducible results over long periods of time (up to 8 hr.).
J. N. ASHLEY

1010. **Ascaridole studies. III. The purification and characterisation of ascaridole.** A. H. Beckett, M. Donbrow and G. O. Jolliffe (*J. Pharm. Pharmacol.*, 1955, **7** [1], 55–65).—Physical constants are given for pure ascaridole obtained by fractional recrystallisation and by chromatography. The use of *m*-dinitrobenzene as a stable secondary standard is recommended in the polarographic assay of ascaridole (*cf.* Beckett and Donbrow, *Brit. Abstr. C*, 1953, 26).
A. R. ROGERS

1011. **The influence of quaternary ammonium compounds in the assay of tyrothricin antibiotics.** L. L. Nisonger (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [12], 716–718).—Cetylpyridinium chloride (**I**), often used as a solubilising agent, has a potentiating effect in the turbidimetric serial dilution assay of methylolgramicidin (**II**), when *Streptococcus* sp. Lancefield group D, strain No. $H69D_5$ is used as the test organism. The effect is represented by the formula $y = 1{\cdot}086 + 0{\cdot}1409\ x$ where y represents the **II** potency ratio and x the ratio of **I** to **II**.
A. R. ROGERS

1012. **Estimation of the concentration of erythromycin in body fluids following intravenous administration.** M. Piller and A. Bernstein (*Schweiz. med. Wochschr.*, 1955, **85** [5], 104–108).—Erythromycin can be estimated by a modification of the method previously described for Aureomycin by Whitlock *et al.* (*J. Lab. Clin. Med.*, 1949, **34**, 1682). It is applicable to serum, urine and bile. The fluid is tested in various dilutions for its bacteristatic effect on test organisms, the inhibition being estimated turbidimetrically. The test organism *Staphylococcus aureus* (Oregon J. No. 9801) is suitable.
G. W. CAMBRIDGE

1013. **The isolation and identification of barbiturates.** F. J. Sabatino (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 1001–1004).—For the identification of barbiturates by means of a paper-chromatographic procedure, 0·5 *M* Na_2CO_3 is used as the stationary solvent, ethylene dichloride as the mobile solvent and ethanolic $AgNO_3$ as the development reagent. R_F values are: phenobarbitone 0·02, butobarbitone 0·18, amylobarbitone 0·35, pentobarbitone 0·47 and quinalbarbitone 0·56. In a column partition method, in which the barbiturate mixture (with formamide and water on Celite) is first eluted with *iso*octane and chloroform (1 : 1), phenobarbitone can be efficiently separated by elution with chloroform and determined by ultra-violet spectrophotometry.
A. A. ELDRIDGE

1014. **A simple method for the estimation of benzocaine in ointments.** F. Michal and R. H. Thorp (*J. Pharm. Pharmacol.*, 1955, **7** [1], 39–41).—A spectrophotometric method is described for the estimation of benzocaine in Compound Ointment of Benzocaine B.P.C. In comparison with the official titrimetric method, there is considerable saving in time and slight improvement in reproducibility. *Procedure*—Warm an amount of ointment containing $\simeq$ 50 mg of benzocaine with light petroleum (boiling range, 50° to 60° C) (100 ml), filter and extract the filtrate with successive 20-ml portions of 10 per cent. HCl soln. Bulk the acid solutions and adjust to 100 ml with 10 per cent. HCl soln. Dilute ten times with water and measure the extinction at 271 mμ against 1 per cent. HCl soln. as blank. The logarithm of the mol. extinction coeff. of benzocaine in 1 per cent. HCl at 271 mμ is 3·065.
A. R. ROGERS

1015. **The paper-electrophoretic identification of *p*-aminobenzoic acid in novocain [procaine] and of *m*-aminophenol in 4-aminosalicylic acid.** G. Wagner (*Die Pharmazie*, 1954, **9** [7], 556–558).—Separation of benzocaine, procaine and *p*-aminobenzoic acid (PAB) and of 4-aminosalicylic acid (PAS), *m*-aminophenol (MAP) and 5-aminosalicylic acid may

be readily obtained by paper electrophoresis in buffers of different pH values. At pH 2·0 benzocaine travels rapidly as a cation, but at higher pH values it migrates only slowly; procaine behaves as a cation between pH 2·0 and 9·0; PAB is amphoteric, the isoelectric point being between pH 3·0 and 4·0, behaving as a cation at pH 2·0 and as an anion between pH 5·0 and 9·0. Similarly MAP is cationic between pH 2·0 and 4·0 and migrates little between pH 5·0 to 9·0; PAS is weakly cationic at pH 2·0 and anionic between pH 4·0 to 9·0; 5-aminosalicylic acid behaves as a cation at pH 2·0 and as an anion between pH 6·0 to 9·0. Identification of PAB is made by diazotising and coupling with alkaline 2-naphthol soln., and of MAP by coupling with diazotised sulphanilic acid and spraying with KOH soln. In the electrophoresis the test soln. is placed in the middle of the filter-paper between the two electrodes. H. F. W. KIRKPATRICK

1016. **Polarographic determination of Intercaine [amethocaine].** Novotný Bohumil (*Českosl. Farmac.*, 1954, **3** [1], 12–13; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 27,594).—Intercaine (amethocaine) is nitrosated and the resulting nitrosamine is determined polarographically by the increment method. For the nitrosamine, $E_{\frac{1}{2}} = -1{\cdot}7$ V (relative to the S.C.E.). The presence of adrenaline does not affect the determination. For $1{\cdot}21 \times 10^{-4}$ to $2{\cdot}42 \times 10^{-4}$ *M* concn. of nitrosamine, the height of the wave is proportional to concn. The precision of the method is ± 1 per cent. E. HAYES

1017. **Determination of aspirin and acetophenetidine [phenacetin] in presence of caffeine by non-aqueous titration.** E. G. Wollish, R. J. Colarusso, C. W. Pifer and M. Schmall (*Anal. Chem.*, 1954, **26** [11], 1753–1754).—For the determination of aspirin in A.P.C. tablets, the constituents are extracted from the tablet mass in acidic medium with $CHCl_3$ - ether (1 + 3) solvent mixture. The solvent is evaporated, the residue is dissolved in dimethylformamide and the aspirin is titrated with 0·1 *N* lithium methoxide in benzene - methanol solvent in presence of thymol blue indicator. The determination of phenacetin is carried out by hydrolysis to phenetidine hydrochloride, extraction of the base from alkaline soln. with $CHCl_3$ and potentiometric titration with $HClO_4$. In the aspirin determination none of the other constituents of A.P.C. tablets interferes, with the exception of stearic acid, which causes a slight positive error. Caffeine may be determined by the method of Wirth on a separate sample aliquot. Methods for the determination of either codeine salts or antihistamines present as additives in A.P.C. tablets are also presented. Recoveries of aspirin from tablets ranged from 99·8 to 100·2 per cent. with a standard deviation of 0·19 per cent., and recoveries for phenacetin ranged from 98·7 to 100·6 per cent. with a standard deviation of 0·8 per cent. G. P. COOK

1018. **The qualitative and quantitative determination of Analgin (sodium 2:3-dimethyl-1-phenylpyrazol-5-on-4-yl-N-methylaminomethanesulphonate).** L. I. Rapaport and M. M. Shvartsburd (*Aptechnoe Delo*, 1954, **3** [5], 47–52).—Two new identity tests for Analgin are proposed. In the first, 0·005 to 0·01 g of Analgin is added to heated H_2SO_4 containing a trace of salicylic acid, when a red colour is formed on the crystals. In the second test, 5 to 6 drops of 20 per cent. *p*-dimethylaminobenzaldehyde in H_2SO_4 (sp. gr. 1·84) are added to 1 ml of a 1 per cent. aqueous soln. of Analgin; an intense yellow colour is produced which does not disappear on the addition of 5 ml of water. The tests are used to detect Analgin in various pharmaceutical mixtures. *Quantitative determination*—(*i*) The sample (0·15 g) is dissolved in 10 ml of water, and titrated slowly with 0·1 *N* iodine soln. after one drop of 0·15 per cent. methylene blue soln. has been added. At the end-point, the colour changes to green (stable for 15 to 20 sec.) and the soln. becomes cloudy. Results on various mixtures are within 1 per cent. of the amount added. (*ii*) The sample (0·13 to 0·16 g) is dissolved in 0·5 ml of water in a 20 to 25-ml glass-stoppered tube; ethanol (5 ml) and chloroform (2 ml) are added and the soln. is titrated slowly with 0·1 *N* iodine chloride in HCl until the rose-violet colour of the upper layer and the brown colour of the lower layer disappear. For determinations on mixtures, the results are accurate to within 1·5 per cent. The chemistry of the reactions and the effects of various interfering substances are discussed. E. HAYES

1019. **Polarographic and potentiometric estimation of 2:3-dimercaptopropanol (BAL) in pharmaceutical preparations.** R. Zumanova and P. Zuman (*Die Pharmazie*, 1954, **9** [7], 554–556).—*Polarographic estimation*—About 0·2 ml of injection soln. (100 mg per ml) of 2:3-dimercaptopropanol (dimercaprol) (**I**) is accurately weighed out, 10 ml of $CHCl_3$ are added and the solution is diluted to 100 ml with ethanol. One ml of this soln. is pipetted into 1 ml of *M* acetate buffer (pH 4·7) and diluted to 20 ml with ethanol. After removal of oxygen the soln. is polarographed and the height of the step that appears at − 0·6 V is compared with standards within the range 0·5 to 1·5 $M \times 10^{-4}$ of **I** in the same electrolyte.

Potentiometric estimation—About 1 ml of injection soln. is weighed out and dissolved in 20 ml of a mixture of equal parts of $CHCl_3$ and ethanol and about 1 ml of pyridine are added. The potentiometric titration is then made with 0·1 *N* iodine with a platinum electrode and a calomel reference electrode. A blank titration is also made. 1 ml of 0·1 *N* iodine ≡ 0·00621 g of **I**. H. F. W. KIRKPATRICK

1020. **Spot-reactions in tablet identification.** P. Cooper (*Pharm. J.*, 1954, **173**, 481–482).—The colour reactions of a long series of drugs of different types with five reagents are tabulated, enabling the probable nature of an unknown tablet to be ascertained. The tests are not necessarily conclusive and confirmatory tests should be made. The reagents used are: (*a*) pyridine - $CHCl_3$ (1:9, by vol.) followed by 0·5 per cent. aq. Cu^{II} acetate, (*b*) aq. 10 per cent. $NaNO_2$ + HCl, (*c*) 20 per cent. aq. HIO_3, (*d*) 1 per cent. aq. $FeCl_3$ and (*e*) 2 per cent. *p*-dimethylaminobenzaldehyde in 20 per cent. HCl. The test is made by addition of one drop of reagent [in the case of (*a*), 2 drops of the solvent mixture followed by 1 drop of the Cu solution] to a small amount of crushed tablet on a piece of filter-paper After 1 min., excess of powder is removed and the colour is observed by light transmitted through the paper. S. C. I. ABSTR.

See also Abstracts 889, 976, 1047, 1048.

Food

1021. **Application of a potentiometric rotary viscometer to measuring consistency of food purées and pastes.** R. J. McColloch and E. A. Beavens (*J. Agric. Food Chem.*, 1954, **2** [19], 986–990).—A potentiometric rotary viscometer that has two types of rotors (one a straight-sided fork and the

other a wire spiral) activated by a synchronous motor through a torsion spring is described for measuring the consistency of food pastes and purées. Displacement of the spring is indicated in degrees of voltage drop, or current flow, which have been calibrated in centipoises against solutions of known viscosity. Results agree well with subjective assessments and the apparatus largely overcomes problems associated with the heterogeneous and thixotropic nature of pastes and purées. S. C. JOLLY

1022. **Plant starch analysis. Rapid determination of starch in apples.** G. H. Carter and A. M. Neubert (*J. Agric. Food Chem.*, 1954, **2** [21], 1070–1072).—A rapid colorimetric procedure that has been developed for determining starch in apples in the maturity range of commercial harvest and storage, is described. It is a modification of the method developed by Pucher and Vickery; perchloric acid is used as a starch solvent. The method was found to be accurate for the following varieties: Jonathan, Golden Delicious, standard Delicious and Winesap. A quarter of the apple starch in these varieties appears to be amylose. A single starch determination takes 15 min., when a 5-min. digestion is used. O. M. WHITTON

1023. **The acid inversion of cane sugar.** E. C. Wood (*Analyst*, 1954, **79**, 779–780).—In several published methods based on the Clerget procedure for determining sucrose, inversion is effected by the addition of a measured vol. of 6·34 *N* HCl. The origin of this strength of acid has been traced to the use by Jackson *et al.* (*Sci. Pap. Bur. Stand.*, 1920, **16**, 141) of a (1 + 1) dilution of some unusually concentrated HCl that they had available. The effect of a variation of 10 per cent. in the acid strength has a negligible effect on the destruction of invert sugar during inversion. The direct effect of a 10 per cent. variation in the amount of acid present on the observed rotation is larger and alters the rotation of the normal wt. of invert sugar in 100 ml by 0·125 international sugar degrees (Jackson *et al.*, *loc. cit.*). These facts indicate that, except in most precise work, the strength of the acid used and the vol. taken can be varied by (say) 5 per cent. without effect on the results. This has been verified experimentally with solutions containing sucrose and lactose and with solutions prepared from condensed milk by the Society of Public Analysts' method. Thus in ordinary analytical work the strength of the acid used may have a tolerance of at least ± 2 per cent. and the vol. added can be measured with a cylinder. A. O. JONES

1024. **Ultramicro-technique for enzymatic hydrolysis of sugars prior to chromatogram analysis.** W. L. Porter and N. Hoban (*Anal. Chem.*, 1954, **26** [11], 1846–1848).—A method is described in which 0·01-ml samples of solutions containing 20 to 30 μg of sugar can be enzymatically hydrolysed in melting-point tubes and the entire sample can then be deposited on paper for chromatographic resolution. The micro-technique required is illustrated with diagrams. G. P. COOK

1025. **The determination of sucrose in sweetened condensed milk: a simplification.** E. C. Wood (*Analyst*, 1954, **79**, 780–781).—A method is indicated for simplifying the arithmetical work in the final calculation of the amount of sucrose in condensed milk determined by the Society of Public Analysts' method. The simplification is effected by taking an accurately weighed amount of the original sample, this wt. depends upon the fat and protein contents of the sample and is ascertained from a table. For many purposes a further simplification can be effected by assuming the protein content to be equal to the fat content, thereby avoiding determination of the former. Another table gives the initial wt. to be taken of a sweetened condensed separated milk for the same simplification to obtain. A. O. JONES

1026. **Standardisation of sieves and determination of grain size of granulated sugar.** J. R. Johnson and J. S. Newman (*Anal. Chem.*, 1954, **26** [11], 1843–1846).—Two methods for the standardisation of sieves, involving the use of standard reference materials for which shape and size distribution are known, are described. With sieves standardised by one of the above methods, a procedure is given by which information about the average grain size and uniformity of grain size of crystalline products, such as sugar, may be obtained. G. P. COOK

1027. **Recent developments in white-sugar colorimetry.** T. R. Gillett and W. D. Heath (*Anal. Chem.*, 1954, **26** [11], 1780–1784).—The proposed method for determining colour involves measurement of the sugar soln. at 420 and 720 mμ and subsequent calculation of the colour index. A standard deviation of ± 1·0 colour index unit was obtained from four samples of sugar analysed by six laboratories. Determinations on a new type of photo-electric colorimeter designed specifically for colour determination on white-sugar soln. give standard deviations of colour index of ± 0·6 units. G. P. COOK

1028. **Chromatographic detection of benzoyl peroxide [in flour].** G. Bionda (*Ann. Falsif.*, 1954, **47**, 174–176).—Methods for the determination of benzoyl peroxide in flour are reviewed and a method for improving the sensitivity of the colour reaction proposed by Calo' and Muntoni (*Rend. Ist. San. Pubbl.*, 1938, **1** [1]) is suggested. *Procedure*—Extract the sample (containing 10 to 50 μg of benzoyl peroxide) with light petroleum and adjust the volume to 1·5 to 7·5 ml. Add 2·5 ml of a 0·1 per cent. solution of dimethyl-*p*-phenylenediamine hydrochloride in ethanol or ethanol - ether. Shake and set aside for 2 to 3 min. Chromatograph on an alumina (neutral or anionotropic) column 4 to 5 mm in diameter. An appreciable colour is given by 20 μg of benzoyl peroxide, but 10 μg can be detected. It is necessary to prepare a reagent blank. G. B. THACKRAY

1029. **Amylograph standardisation.** Amylograph Standardisation Committee of the American Association of Cereal Chemists. (*Trans. Amer. Ass. Cereal Chem.*, 1954, **12** [3], 292–299).—A progress report embodying a study of the instruments and procedures in use is presented. In comparison with current routine procedures, the tentative standard method described for determining max. viscosities attained by flour and buffer mixtures with rise in temp. gives closer agreement between collaborative results. P. S. ARUP

1030. **The determination of lactose in milk changed by lactic fermentation. The conditions for quantitative hydrolysis of the lactose in whey. II.** I. Saenz Lascano Ruiz (*Ann. Falsif.*, 1954, **47**, 285–305).—Glucose, galactose and lactose are separately detected and determined by paper-partition chromatography by use of the organic phase of the mixture *n*-butanol - acetic acid - water (40:10:50 by vol.) as solvent, the spots being revealed on

spraying with aniline phthalate reagent and heating to 100°C. The whey is prepared for chromatography by coagulation with acetic acid or rennet, but the latter serum (as does whole milk) gives elongated spots. For quant. estimation, known reference samples are chromatographed at the same time. By this means hydrolysis curves at various pH, times and temp. are prepared; sulphuric acid is used as a hydrolysing agent since it is easily eliminated with $Ba(OH)_2$ before chromatography. From the results, a technique for the hydrolysis is obtained in which the milk is coagulated and the whey is hydrolysed at a pH of 0·2 to 0·25 for 4 hr. over boiling water. The amount of glucose and galactose is determined by titration with 0·1 *N* $KMnO_4$ (conversion tables given), and the lactose figure is corrected by an addition for any lactic acid present. The use of the method on fresh, pasteurised and preserved milks is discussed. E. J. H. BIRCH

1031. **Identification of insect setae as an index of contamination in dairy products.** F. J. Sabatino (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 960–966).—Furrowing, branching or feathering and pigmentation are useful characteristics for identification of fly setae. Pigmentation of setae in milk persists during the manufacture and analysis of butter. The shape of the base of the setae may also be characteristic. Typical setae are figured.
A. A. ELDRIDGE

1032. **Freshness test of tinned fish meats by paper chromatography.** T. Yamanishi, S. Hanai, K. Fukuhara and C. Inagaki (*Natural Science Report, Ochanomizu Univ.*, 1954, **5** [1], 101–108).—The histamine content of tinned fish can be a good criterion for the test of freshness. Tinned fish meat (20 g) is ground in a mortar with pure sand (5 g) and water (40 ml). After 30 min., the paste is extracted with 6 *N* HCl (5 ml) and centrifuged. The supernatant liquid (25 ml) is evaporated to 10 ml and one drop (0·0025 ml) of it is submitted to paper chromatography, by making use of *n*-butanol saturated with 10 per cent. aq. NH_3 as solvent. The spot is revealed by ninhydrin (0·25 per cent. in acetone) and the elliptic area measured. (R_F 0·53). The efficiency of extraction was examined by the Rose - Lubuschez method (*J. Biol. Chem.*, 1950, **183**, 731) and found to be 98 per cent. The water content of fish meat is assumed to be 75 per cent. When the area of the spot (sq. cm) is plotted against the log. of the concn. of histamine dihydrochloride (g per ml), a straight line is obtained within 250 to 5000 μg per ml (18 to 360 mg of histamine per 100 g of meat). As the lower limit of histamine in meat causing poisoning is approx. 100 mg per 100 g of meat this method is suitable for practical uses. Several results with different fish were compared with the Rose - Lubuschez method and the agreement between them was found satisfactory. The histamine contents of various fish of different freshness were measured. They are parallel to the contents of trimethylamine or of volatile nitrogen, which are usually considered to be the criteria of freshness of fish.
K. SAITO

1033. **Partition chromatography of fatty acids.** J. Spiteri (*Bull. Soc. Chim. Biol.*, 1954, **36** [9], 1355–1362).—Fatty acids (C_{10} to C_{24}) are separated by partition chromatography on paper impregnated with liquid paraffin and with glacial acetic acid as the moving phase. Impregnation is carried out by vertical capillary absorption of the liquid paraffin (10 per cent. in benzene). The chromatogram is then run in the same direction, as there is a progressive concentration of the oil from the base upwards. Impregnation of the paper by dipping in the paraffin solution results in erratic R_F values. The chromatogram is allowed to dry, and as soon as it becomes opaque it is placed flat in a dish of water, which removes much of the acetic acid and paraffin oil. The liquid is poured off and replaced with the min. amount of ammoniacal 1 per cent. $AgNO_3$ soln. After 10 to 15 min., the soln. is poured off and the chromatogram is washed, and then treated with very dil. ammonium sulphide, which is replaced when the spots begin to appear. The positions of the acids, which show as light brown to black spots on a white background, are marked as soon as the chromatogram is washed. Results obtained with the acids of an olive oil are recorded.
C. E. SEARLE

1034. **The estimation of unsaturated fatty acids by reversed-phase partition chromatography.** W. M. L. Crombie, R. Comber and S. G. Boatman (*Biochem. J.*, 1955, **59** [2], 309–316).—The separation of representative unsaturated acids on a standard reversed-phase column is investigated. A method is described for the analysis of 1 to 60 mg of mixtures of saturated and unsaturated fatty acids. The column consists of water-repellent kieselguhr impregnated with liquid paraffin. The acids are eluted by mixtures of acetone and water. Portions of eluate are titrated under nitrogen with 0·01 *N* KOH in methanol added from a 0·5-ml Agla micrometer syringe; titrations to within 0·001 ml are made accurately. The most widely distributed unsaturated fatty acids are separable from each other by the described method, but not from some saturated acids that are eluted at similar rates. These saturated acids are determined by a second chromatogram after oxidation by alkaline $KMnO_4$, which oxidises the unsaturated acids; the amounts of these are then ascertained by difference. The results agree with those obtained by other methods. The effects on rate of elution of substituents and of type and number of unsaturated groups in the acids are determined.
J. N. ASHLEY

1035. **Methods for evaluating the relative effectiveness of the determinations used in the analysis of maple products.** A. Cholette and M. Jean (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 939–945).—Mathematical and graphical procedures for assessing the relative efficacy of methods of analysis of maple syrup are discussed and applied. The conclusions are subject to revision in the light of further analytical information. For adulteration with brown sugar, the P_2O_5 value is the most significant, whilst for white sugar, the K_2O value is preferable. For brown sugar, conductivity is more reliable than malic acid value. A graphical evaluation permits the method to be applied to mixtures containing two adulterants.
A. A. ELDRIDGE

1036. **Determination of bromoacetic acid and its esters in beverages.** G. Curli and V. Prati (*Chim. e Ind.*, 1954, **36** [9], 704–705).—The standard method of Florentin and Munsch (*Ann. Falsif.*, 1936, **29**, 104) gives low results, owing to the high solubility of bromoacetic acid and its ethyl and benzyl esters in water and to their volatility. The following method gives results only slightly below theoretical values for beverages containing about 2 mg of organic Br per litre. Wine or other beverage (100 to 200 ml) is extracted with five 30 to 60-ml portions of ether, the extract is filtered, the filter-paper is washed with ether, and the filtrate plus

washings are boiled gently under reflux with 5 ml of 0·25 *N* bromine-free NaOH for 1 hr. The ether is then distilled off, and the aqueous residue is added to 0·5 g of bromine-free MgO in a crucible. The mixture is evaporated to dryness on a water-bath and the residue is calcined at a temp. below red heat. The calcined residue is then extracted repeatedly with boiling water, the vol. of extract is made up to 25 ml and Br′ is determined in aliquots by Dénigès' method. R. TRUSCOE

1037. **The determination of lead in cocoa with a square-wave polarograph.** D. J. Ferrett, G. W. C. Milner and A. A. Smales (*Analyst*, 1954, **79**, 731–734).—The rapid determination of < 1 p.p.m. of Pb in a sample of cocoa is described. *Procedure*—The sample (2 to 3 g) is ashed at 500° C, the residue is dissolved in HCl and the Pb in the soln. is determined directly by means of a square-wave polarograph. The blank result in this procedure is equivalent to 0·05 p.p.m. or less of Pb. When the organic matter was destroyed by wet oxidation with HNO_3 and $HClO_4$ the same amount of Pb was obtained; hence no loss of Pb by volatilisation at 500° C occurs. The polarographic determination of Pb in the residue from the dry-ashing procedure avoids the high blanks of the chemical methods. A. O. JONES

1038. **A rapid method for the estimation of copper in cider.** C. F. Timberlake (*Chem. & Ind.*, 1954, [47], 1442–1443).—The Cu content of a cider may be quickly and accurately determined by extracting the cider, acidified to *N* with H_2SO_4, with a solution of zinc dibenzyldithiocarbamate in CCl_4 without either acid wet-digestion or initial heating, provided that the cider is free from deposit or suspended matter. Two spectroscopic methods, one being applicable to a large number of samples, and one visual method are given. They are applicable to other liquid products. O. M. WHITTON

1039. **Importance of paper chromatography in brewing technological research. I. Technique.** A. Stöckli (*Schweiz. Brauerei Rdsch.*, 1954, **65** [9], 139–144).—Descriptions are given of the principles of one- and two-dimensional chromatography, and of a technique in which the unsprayed spots, including incompletely separated constituents, are separately cut out and sewn on to new paper strips for development with suitable solvent mixtures. An example is given of the separation of a mixture of twenty-two amino-acids. P. S. ARUP

1040. **Contribution to the objective evaluation of the aroma of hops.** V. Salač (*Brauwissenschaft*, 1954, [6–10], 117–123, 147–149, 169–176, 197–200, 217–222).—Conventional physical and chemical tests are insufficient for evaluation of hop aroma and in particular there exists no method for the objective characterisation of the aroma, owing to the complexity of the fragrant substances. The often unpleasant garlic-like component is attributed to organic sulphur compounds, but there seems to be no relationship between the intensity of this component and the total S in the ether extracts of various hop species, as established by the Grote - Krekeler method. In an attempt to isolate individual sulphur compounds an apparatus for the extractive steam distillation of crushed hops was developed. The turbid distillate was clarified with methanol, and di-allyl sulphide was found by the pink coloration formed with phloroglucinol - HCl. The allyl sulphide contents in hops of various origin was established by using a Lange colorimeter with 20 per cent. absorption and with the help of colour filters. The colorations were compared with those obtained with pre-determined solutions of pure di-allyl sulphide. It was further established that the di-allyl sulphide was a product of the reaction of linalol or its esters with SO_2 used in the sulphurisation of the hop plants. The smallest amount of di-allyl sulphide was found in the high-grade hops of Saaz origin. Numerous conventional analytical tests with special emphasis on acetyl and iodine numbers were carried out with hop oil quant. obtained by treating the turbid steam-distillate with active carbon, extracting the dried carbon with ethanol and evaporating the latter. The differences in iodine numbers are not typical for the smell of particular hops species. The percentage of high-boiling substances in hop oil which influence the taste of beer was determined by evaporation of hop oil in a carbon dioxide atmosphere at 70° to 100° C. Although the lowest content of non-volatile substance again was established in hops of Saaz origin, no clear relationship could be found between the commercial gradation of hops and the amounts of non-volatile substance in the respective oils. H. BURSTIN

1041. **Determination of extract and cold water extract in malt by means of the refractometer.** R. E. Essery (*J. Inst. Brewing*, 1954, **60** [4], 303–306).—Measurements of refractive index can replace sp. gr. in the determinations of extract and cold-water extract on pale malts. The method is not suitable for coloured or black malts. A mash is prepared according to the standard method, and five readings are taken with a Zeiss immersion refractometer at 20° ± 0·1° C (previous filtration is unnecessary). Tables given by the A.O.A.C. ("Methods of Analysis," 7th Ed., Washington, D.C., 1950, 41·9) give values of *n*, which are in turn converted to extract (lb per qr.) or to cold water extract from tables. The method has a standard deviation of ± 0·66 lb per qr. for the extract and ± 0·47 per cent. for the cold water extract. G. B. THACKRAY

1042. **Chromatographic determination of glutamic acid in distillery residues.** F. Parisi and G. Funes (*Chim. e Ind.*, 1954, **36** [9], 684–692).—Acid Duolite C-3 resin is prepared by washing successively with 6 per cent. aq. NH_3, water, 10 per cent. HCl and water. A diluted HCl hydrolysate (pH $\lessdot$ 1·84) containing HCl, 0·05 per cent., glutamic acid, 0·016 to 0·048 per cent., and inorganic material 0·1 to 0·14 per cent. is passed through a column of resin, with the eluate remaining at pH $\lessdot$ 1·4; the resin is washed with water and eluted with 300 ml of 3 per cent. aq. NH_3, and the eluate is concentrated in a vacuum, with the exclusion of O, at $\gtrdot$ 50° C. The vol. is made up to 100 ml and aliquots (pH $\gtrdot$ 8·5) are passed through an acid alumina column (activated by soaking in *N* HCl, followed by washing until the effluent has a pH $\simeq$ 3·5); the column is washed with successive small portions of water to remove amino-acids other than glutamic and aspartic acids, which are then eluted with 150 ml of 6 per cent. aq. NH_3. The eluate is evaporated down, as before, to about 20 ml, the vol. is made up to 50 ml, the solution is filtered and an ascending paper chromatograph is taken, with the solvent pyridine - acetic acid - H_2O (50:35:15), using ninhydrin for development; the intensity of the spots is compared with those given by known concn. of glutamic acid. The glutamic plus aspartic acid content of a number of samples varied from 6·5 to 11·8 per cent. R. TRUSCOE

1043. **Standards for the evaluation of kieselguhr.** K. Kärnbach (*Die Brauerei*, 1954, **8** [101–102], 648–654).—The kieselguhr-filter is recommended for the clarification of beer, but careful standardisation is necessary. Analysis of the kieselguhr may be limited to the following essential points: loss on ignition, water content, proportion of heavy particles, and rate of filtration, and effect on smell, taste, colour, pH and iron content of beer.
A. R. ROGERS

1044. **Metallic elements in wine by flame photometry.** M. J. Pro and A. P. Mathers (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 945–960).—A technique for determining metallic elements in wine in which a Beckman Model DU flame spectrophotometer and an oxygen - hydrogen flame are used is described. Determinations of Na, K, Ca and Mg in filtered wines checked for Ca and Mg by chemical methods show that these elements can be determined accurately by flame photometry. Interferences were investigated; < 5 per cent. of sucrose has no effect, whilst ethanol enhances light emission. Most of the cations examined affected emission by Ca and Mg, but only Li affected that by Na or K, *e.g.*, Fe (> 2 p.p.m.) and Mn (> 10 p.p.m.) inhibited light emission by Ca and Mg. Phosphate (> 500 p.p.m.) depressed light emission by K but not that by Na. The interfering effect of phosphate and sulphate is minimised by the addition of dextrose.
A. A. ELDRIDGE

1045. **Simplified separation of non-saponifiable matter in the analysis of oils.** F. J. Mulder (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [8], 626–628).—The non-saponifiable matter (*e.g.*, containing vitamins A and D) in the oil sample is separated by saponification of the oil (0·1 to 1 g) in *N* methanolic or ethanolic KOH (10 ml), followed by the addition of a known vol. ($\simeq$ 40 ml) of dry benzene to the alcoholic soap soln. The benzene soln. is then washed several times with H_2O ($\simeq$ 25 ml), dried over flake $CaCl_2$ and filtered; the concn. of non-saponifiable matter is determined by evaporating the benzene and drying and weighing the residue. The method, which is applicable to food preparations, obviates the usual lengthy extraction and the quant. transfer of the extracts.
W. J. BAKER

1046. **Thiamine - pyramin assay. Factors influencing the yeast fermentation methods for thiamine and Pyramin.** W. O. Caster and O. Mickelsen (*J. Agric. Food Chem.*, 1954, **2** [21], 1073–1076).—The effect of a number of physical and chemical factors on the yeast fermentation method for thiamine and pyramin has been studied. Among the potential sources of error in the pyramin method are the effects resulting from the addition of excess of peroxide in the sulphiting step, inadequate shaking during the fermentation period and the stimulatory action of 5-β-hydroxyethyl-4-methylthiazole. It is shown that if the sulphite blank activity exceeds that of the 1-μg thiamine standard the usual addition of 1 μg of thiamine to the blank should be omitted.
O. M. WHITTON

1047. **Determination of vitamin B_6 in pharmaceutical preparations.** Lajosné Szabolcs (*Magyar Kém., Fóly.*, 1953, **59** [7], 218–220; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 31,134).—Dissolve a sample containing 0·02 g of pyridoxine hydrochloride in 150 ml of water in a 500-ml conical flask, add 20 ml of 0·1 *N* $KBrO_3$, $\simeq$ 0·2 g of KBr and 10 ml of 15 per cent. HCl; set aside in the dark for 45 min. Add 0·2 g of KI and, after 5 min., titrate the liberated iodine with 0·1 *N* $Na_2S_2O_3$. Other vitamins of the B group, such as thiamine and riboflavin, do not interfere, but erroneous results are obtained if photo-decomposition products of riboflavin are present.
E. HAYES

1048. **The analysis of analogues. A development of the general theory of partition and its application to the determination of cyanocobalamin and hydroxocobalamin in mixtures.** J. G. Heathcote and P. J. Duff (*Analyst*, 1954, **79**, 727–731).—Theoretical considerations show that for a given solvent to water ratio there is a unique relationship between the composition of a mixture of analogues and the apparent partition coeff. as measured by some common property. In the method described, the determination of the apparent partition coeff. is based upon the spectrophotometric determination of the total cobalamin in each phase of a benzyl alcohol and water partition at the wavelength (356 mμ), at which both components have the same specific extinction, *viz.*, 174. The total cobalamin concn. in each phase is given by $\frac{\text{E at } 356}{0{\cdot}0174}$ μg per ml and the apparent distribution coeff. for any particular mixture is given experimentally by $K = \frac{\text{E at } 356 \text{ (solvent)}}{\text{E at } 356 \text{ (water)}}$. With mixtures of cyanocobalamin and hydroxocobalamin of known composition results were good.
A. O. JONES

1049. **Thiocyanogen bromide method for the determination of nicotinic acid.** V. M. Iosikova (*Tr. Vses. Vitaminnogo In-ta*, 1953, **4**, 226–230; *Referativnŷĭ Zh., Khim.*, 1954, Abstr. No. 29,346).—The sample (1 to 3 g) is hydrolysed by heating it to 100° C for 1 hr. with 100 ml of *N* HCl; the hydrolysate is neutralised to pH 6·5 with NaOH and 100 ml of ethanol are added. The nicotinic acid is then adsorbed on Askanite; the adsorbate is extracted with 25 ml of *N* NaOH and the resulting soln. is centrifuged. Impurities in this soln. are removed by treatment with saturated $ZnSO_4$ soln. (pH 6·5) and *N* $KMnO_4$ soln.; the excess of Zn is then removed with potassium phosphite and the purified extract is treated with BrCNS (2 ml BrCNS plus 7 ml of ethanolic soln. of aniline heated to 60° to 70° C for 30 to 60 min.), The colour is measured absorptiometrically.
E. HAYES

1050. **Determination of arachidonic, linolenic and linoleic acids (vitamin F) in milk and blood. II. Determination in milk.** W. Pasquali (*Acta Vitaminol.*, 1954, **8** [3], 113–120).—The milk (5 ml) is shaken with absolute ethanol (25 ml) and 30 per cent. aq. NaOH (2 ml) and boiled under reflux for 20 min. The ethanol is removed and the fatty acids are extracted with ether after acidification. The dried fatty acids are heated with KOH in ethylene glycol (7·5 g of KOH in 1·2 ml of H_2O diluted to 100 ml with ethylene glycol) at 180° C for 30 min. in order to isomerise their double bonds to conjugated double bonds. The fatty acids are then isolated and dissolved in absolute ethanol and the absorption is measured at 316 mμ (arachidonic acid), 268 mμ (arachidonic acid + linolenic acid) and at 234 mμ (sum of the three acids). The average error in single measurements is for linoleic acid, $\pm$ 2·5 per cent., for linolenic acid, $\pm$ 25 per cent. and for arachidonic acid, $\pm$ 12 per cent.
N. E.

See also Abstract 889, 898, 942.

Sanitation

1051. **Analysis of bore-hole water.** M. Killer (*Nafta, Zagreb*, 1954, **5** [7], 187–190).—Standard turbidimetric and colorimetric procedures for the determination of SO_4'', NO_3', Fe and silicate are described. R. Truscoe

1052. **A continuous recorder for dissolved oxygen in water.** R. Briggs, G. Knowles and L. J. Scragg (*Analyst*, 1954, **79**, 744–752).—In the apparatus described and illustrated, the Winkler method for determining O in water has been mechanised and adapted to automatic working. The addition of reagents to the water in a reaction cell in the correct order and concn. is controlled by a series of electromagnetic valves and in the final stage the optical density of the liquid containing the liberated iodine is measured at 450 mμ and recorded. A sulphite soln. is then automatically added to remove the iodine and the optical density is again recorded. The O content at any time can then be calculated from the difference between the two recorded readings. With water containing 2 to 8 p.p.m. of O the max. error was 0·05 p.p.m. A. O. Jones

1053. **Method for determination of dissolved oxygen. Application to liquid bacterial cultures.** B. Warshowsky and E. J. Schantz (*Anal. Chem.*, 1954, **26** [11], 1811–1814).—A specially designed amperometric titration cell, with a rotating platinum or gold electrode, that also serves as a bacterial growth chamber, is carefully sterilised, and the substrate (450 ml) containing tributyl phosphate (1 ml) is placed in it. Air is passed in at 300 ml per min. and the current is measured at an applied potential of −0·75 V with respect to the S.C.E. Accidental contamination is monitored by passing the air for 16 hr. and examining the constancy of the current after discontinuation of the air supply. The medium is inoculated and the aeration is continued. Residual current readings are taken periodically after the passage of air has been discontinued and N has been passed at the same speed for 15 min. Oxygen concn. is derived from a linear relationship with diffusion current. Results obtained on soln. of 0·1, 0·5, 1, 2 and 3 *M* NaCl, *M* Na_2SO_4, 0·25 *M* NH_4Cl and *M* $CaCl_2$, and a culture of *Sarratia marcescens* agree with those by the Winkler method; precision is ± 0·2 in 5·9 p.p.m. D. A. Pantony

1054. **Some notes on the determination of dissolved oxygen.** Water Pollution Research Laboratory (*J. Inst. Sewage Purif.*, 1953 [1], 15–22).—Electrometric methods for the titration of iodine solutions give a more precise end-point than the use of a starch indicator. Oxygen determination, by means of a van Slyke apparatus is mentioned. A. Webster

1055. **A respirometer for the study of the oxygen demand of polluted water and sewage.** A. B. Wheatland and R. Lloyd (*Lab. Practice*, 1955, **4** [1], 6–10).—A respirometer is designed for use with large samples of water, air being used as the gas phase. The sample (750 ml) is placed in a 1-litre flat-bottomed flask, which is connected by a ground-glass joint to a mercury differential manometer, on the far side of which is a temp.-compensating bulb immersed in the thermostat with the flask. An absorption tube containing potassium hydroxide is hung in the neck of the flask. Air is admitted to the flask and, after being stirred for 1 hr., pressure readings are taken at intervals; when the pressure difference reaches 3 cm, O is admitted to the flask from a gas-pipette. Detailed instructions are given for calibrating the apparatus, and sources of error are discussed. A typical oxygen demand - time curve is given. H. P. Paget

1056. **Micro-analytical method for the determination of sulphur dioxide in the atmosphere.** H. Stratmann (*Mikrochim. Acta*, 1954, [6], 668–678).—The SO_2 is adsorbed initially on silica gel and then reduced with H to H_2S on a platinum contact catalyst at 700° to 900° C. The H_2S and excess of H are passed into 2 per cent. ammonium molybdate in 0·4 *N* H_2SO_4. The resulting blue - violet Mo complex is then determined colorimetrically with the aid of a Zeiss Opton S 57 filter. The Beer - Lambert law is obeyed. According to the amount of the reagent solution used, 1 to 200 μg of SO_2 can be determined. Three hundred litres of air per hr. can be passed over the adsorbent without danger of incomplete adsorption. If the quantity of air used contains > 300 mg of H_2O, it must be dried by passing through a preliminary vessel containing P_2O_5. An excess of pressure up to 200 mm does not affect the adsorption, and variation of temp. between 18° C and 40° C is also without effect. Concentrations of SO_2 of ≃ 0·01 μg per litre of air may be accurately determined. A. J. Mee

1057. **Determination of cyanides in water and waste samples.** F. J. Ludzack, W. A. Moore and C. C. Ruchhoft (*Anal. Chem.*, 1954, **26** [11], 1784–1792).—Three methods were investigated to select an analytical procedure suitable for use as a standard method for cyanide determinations, regardless of sample type, origin or associated interferences. The procedures proposed are: the benzidine - pyridine method of Aldridge as modified by Nusbaum and Skupeko (*Brit. Abstr. C*, 1952, 222); the pyridine - pyrazolone method of Epstein (*Brit. Abstr. C*, 1947, 117); and the modified Liebig titration with *p*-dimethylaminobenzylidenerhodanine as indicator of Ryan and Calshaw (*Analyst*, 1944, **69**, 370). All samples to be analysed for cyanide should be treated by acid distillation except where experience shows otherwise; certain samples containing steam-distillable interferences require solvent extraction with *iso*octane, hexane or chloroform before distillation. The pyridine - pyrazolone colorimetric method is most suitable for cyanide concn. < 1 p.p.m. and the titrimetric method for concn. > 1 p.p.m. For most samples, 90 per cent of added cyanide was recovered and on sewage, river water and samples containing low concn. of interfering compounds that were separated by distillation only, 95 per cent. of added CN′ was recovered. G. P. Cook

1058. **Determination of fluorine in tap-water. Modification of the method of Willard and Horton.** J. Bouman (*Chem. Weekbl.*, 1955, **51** [3], 33–34).—The fluorimetric method of Willard and Horton, based on the decrease of fluorescence of the Al - morin complex in ethanol when F is present, is used, but the concn. are altered to increase sensitivity. The greatest decrease in fluorescence was found when the Al - morin complex was in 10 per cent. ethanol and the pH was 4·4. The F in 0·25 ml of H_2O can be determined with an accuracy of ± 0·02 μg per ml. A. J. Mee

1059. **The use of methyl orange for the determination of residual chlorine in water.** P. M. Litvinenko and V. S. Rozovskiĭ (*Gigiena i Sanit.*, 1954, [1], 52–53; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 27,569).—To 100 ml of water, add 2 drops of 5 *N*

HCl and titrate with 0·005 per cent. methyl orange soln. until a faint rose colour persisting for a minute is obtained. One ml of this soln. corresponds to 0·00219 mg of Cl. The methyl orange soln. is prepared immediately before use by diluting a 0·05 per cent. soln.; the stronger soln. is stable for three months. In contrast to the iodimetric method, the proposed method permits the determination of free Cl, since methyl orange does not react with mineral or organic chloramines. The method is sensitive to 0·02 mg of Cl per litre. E. HAYES

1060. **Controlling factors in identification of [airborne] microscopic chloride particles with sensitised gelatin films.** F. D. Pidgeon (*Anal. Chem.*, 1954, **26** [11], 1832–1835).—The method is essentially a modified spot test. A gelatin film sensitised with mercurous fluorosilicate is prepared. When a chloride particle comes into contact with the sensitised gelatin, it forms a characteristic spot or halo, which can be recognised with a dark-field microscope or, when the spot is large, with the eye. To interpret the observations it is necessary to know the growth and fading ratio of the halos and the relation of halo size to particle size as a function of both temp. and film thickness for a range of halo sizes. The log. of particle size varies linearly with the log. of halo size. The growth rate is independent of the method of collection, the sensitised gelatin mixture, the age of the sensitised film, the mercurous fluorosilicate reagent and variation of temp. No significant effect of thickness on growth rate was found for films of thickness from 24 to 200 μ, but there was a definite decrease in growth rate for films of thicknesses < 18 mμ. Sodium chloride halos fade a little, but this is insignificant when the diameter > 150 μ; this limiting diameter is reduced to 60 mμ when the cardioid dark-field condenser is used. A. J. MEE

1061. **Analytical applications of the hydrolysis kinetics of some organo-phosphorus insecticides.** P. R. Carter (*J. Sci. Food Agric.*, 1954, **5** [10], 457–460).—The analysis of organo-phosphorus insecticides is often closely associated with the hydrolysis of these compounds. The application of the results of the hydrolysis kinetics to the determination of the purity of P-containing insecticides, particularly parathion and Paraoxon, is discussed. For the hydrolysis of parathion and its impurities, only alkaline conditions, in which the *p*-nitrophenoxide ion is produced, are considered. The yellow colour of the *p*-nitrophenoxide produced is compared, at timed intervals, with that from a known concentration of *p*-nitrophenol. The nitrophenol group is almost exclusively removed by hydrolysis and the reaction can be followed colorimetrically to $\not< 90$ per cent. of complete hydrolysis. The reaction is probably bimolecular. The mathematical treatment and experimental details are discussed. R. J. MAGEE

1062. **Paper chromatography of some organic phosphate insecticides. I. New spot test.** J. W. Cook (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 984–987).—Of the organic phosphate insecticides examined, technical Systox, parathion, Methyl parathion, Chlorthion, Malathion, EPN and Diazinon can be detected on paper chromatograms, the whole paper being sprayed with N-bromosuccinimide as brominating agent, and the unabsorbed Br being converted into bromofluorescein by overspraying with fluorescein solution. The fluorescence of unbrominated fluorescein reveals the spot of organic phosphate. A. A. ELDRIDGE

1063. **Paper chromatography of some organic phosphate insecticides. II. Separation and identification.** J. W. Cook (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 987–989).—Separation is accomplished by reverse-phase paper chromatography; mineral oil is used as the stationary phase and ethanol, acetone and water (1:1:2, by vol.) as the mobile phase. R_F values are: Systox unknown component 0·10, Systox isomer 0·47, Isosystox isomer 0·99, EPN 0·35, parathion 0·52, Diazinon 0·61, Chlorthion 0·70, Methyl parathion 0·85 and Malathion 0·93. A. A. ELDRIDGE

1064. **Separation and identification of chlorinated organic pesticides by paper chromatography. VI. Technical benzene hexachloride, Lindane, technical DDT and Rhothane.** L. C. Mitchell (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 996–1001).—A solvent system comprising a vegetable oil (*e.g.*, soya-bean oil) as the immobile solvent and aqueous acetone or aqueous dioxan as the mobile solvent separates BHC, Rhothane and DDT, but not the isomers of BHC and DDT. With dimethylformamide as the immobile solvent and *n*-hexane ($< 25°$ C) or mineral oil ($> 25°$ C) as the mobile solvent, the four principal isomers of BHC can be separated from each other and from DDT, but Rhothane cannot be separated from Lindane. A. A. ELDRIDGE

1065. **An impurity compensated polarographic method for the determination of the gamma isomer in technical benzene hexachloride.** J. Watt (*Analyst*, 1954, **79**, 735–744).—A rapid and precise method for the determination of the biologically active γ-isomer in technical hexachloro*cyclo*hexane is described. The apparent amount of the γ-isomer is first determined by a normal polarographic procedure and a correction for interference by impurities is obtained from a polarographic comparison of two saturated solutions of the γ-isomer, one containing only the pure γ-isomer and the other a known amount of the technical sample together with an excess of the γ-isomer. If an excess of the γ-isomer is shaken with the supporting medium (1 per cent. of KI and 0·005 per cent. of gelatin in a mixture of equal parts of ethanol and water) and the soln. is examined polarographically, a wave is observed of height corresponding to a saturated soln. of the γ-isomer. If a second soln. is prepared by shaking a known small amount of the technical sample and an excess of the γ-isomer with the medium, the height of the wave for the γ-isomer then exceeds that of the first soln. by an amount that varies according to the interfering impurities present. The results agree with those of other methods and the precision of a single determination is ± 5 per cent., expressed as 95 per cent. confidence limits. The determination can be made with a simple and inexpensive manually operated polarograph. A. O. JONES

1066. **Acaricide determination. Micro-determination of the acaricide ethyl *pp'*-dichlorobenzilate (Chlorobenzilate).** R. C. Blinn, F. A. Gunther and M. J. Kolbezen (*J. Agric. Food Chem.*, 1954, **2** [21], 1080–1083).—Two analytical methods are presented for the micro-determination of the acaricide ethyl *pp'*-dichlorobenzilate (**I**) in the presence of citrus extractives. Both methods are based upon the hydrolysis of **I** in the extractive mixture to *pp'*-dichlorobenzilic acid, which is then selectively oxidised to *pp'*-dichlorobenzophenone. This ketone is determined either by its absorption at 264 mμ or by the absorption of its 2:4-dinitrophenylhydrazone in alcoholic alkali at 510 mμ.

Both methods are sensitive to $\simeq$ 15 μg of **I** in admixture with 3 g of citrus extractives. These procedures involve a two-stage clean up of accessory extractives and hence they are very specific. O. M. WHITTON

1067. **Colorimetric reactions of *p*-chlorobenzyl *p*-chlorophenyl sulphide (Chlorbenside) and its sulphone.** D. J. Higgons and D. W. Kilbey (*Chem. & Ind.*, 1954, [44], 1359–1360).—Two colorimetric methods are described for the determination of *p*-chlorobenzyl *p*-chlorophenyl sulphone (Chlorbenside sulphone), which is obtained by the oxidation of Chlorbenside. In the first method, Chlorbenzide sulphone is nitrated and the product is treated in benzene with 10 per cent. sodium methoxide in methanol to give a purple colour, which reaches its max. intensity in 1 to 2 min. and then steadily falls. The second method involves a condensation with *m*-dinitrobenzene and KOH under conditions similar to those used for the colorimetric analysis of substances in urine extracts containing the $\cdot CH_2 \cdot CO \cdot$ group (*Biochem. J.*, 1938, **32**, 1312). Residues of Chlorbenside or its oxidation products are found to interfere seriously with the Schechter - Haller method for the colorimetric analysis of DDT. D. BAILEY

See also Abstract 916.

Agriculture and Plant Biochemistry

1068. **Determination of alkaloids in fodder lupins. II. Gravimetric and colorimetric micro-methods.** Z. Wierzchowski (*Rocz. Nauk. Rolniczych., B*, 1954, **67**, 389–409).—Ground sweet-lupin seed (0·02 to 0·1 per cent. of alkaloid) is extracted with ether - chloroform and the extract is evaporated to dryness in a vacuum. The aq. acid extract of alkaloids in the residue is treated with a 1 per cent. ammonium reineckate soln. After 12 hr. at 4° to 5° C, the ppt. is collected on a glass filter (porosity, 3), washed with water and dissolved in acetone. The intensity of the purple coloration is determined photometrically. Reineckate solutions containing 0·3 to 6·0 mg of alkaloid in 5 ml of acetone follow the Beer - Lambert law. Alternatively, the reineckate ppt. is dried at 101° to 102° C and weighed. The conversion factor (ppt. $\rightarrow$ alkaloid) for yellow lupin is 0·32; for blue lupin in $\simeq$ 1-mg amounts the factor is 0·32 and for larger amounts, 0·34; for white lupin, the corresponding values are 0·30 and 0·31. Crystals of the reineckates of sparteine, lupinine, lupanine and hydroxylupanine are described and photomicrographs are presented.
A. G. POLLARD

1069. **Some observations on the Versenate method for calcium and magnesium in agricultural liming materials.** W. M. Hoffman and H. Shapiro (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 966–971).—Results for the determination of Ca by titration with disodium ethylenediaminetetra-acetate (Versenate) with visual (murexide) or photometric observation of the end-point compared favourably with those obtained by the A.O.A.C. method (*cf.* Schwarzenbach and Ackermann, *Helv. Chim. Acta*, 1947, **30**, 1798). For Mg, differential titration afforded higher values, but direct titration (with Eriochrome black T as indicator) after removal of Ca was satisfactory. A. A. ELDRIDGE

1070. **Ethylenediaminetetra-acetic acid as an analytical reagent to evaluate agricultural liming materials.** A. T. Perkins, J. F. Merrill and J. B. Idleburg (*J. Ass. Off. Agric. Chem.*, 1954, **37** [4], 971–975).—Satisfactory results in the determination of Ca and Mg in various calcareous materials are tabulated. The Ca and Mg may be extracted with acid and then titrated with EDTA, or the mineral may be solubilised with EDTA, the excess of which is then titrated with $CaCl_2$. For determining Ca plus Mg, Eriochrome black T is used as indicator; for determining Ca alone, murexide is employed.
A. A. ELDRIDGE

1071. **The errors involved in pH determination in soils.** M. Raupach (*Aust. J. Agric. Res.*, 1954, **5** [4], 716–729).—The relative accuracy of soil pH determinations is evaluated. Errors of replication shown by variations between routine observers and by variations between determinations on different days, errors due to soil variation and errors within the measuring system, *e.g.*, due to the suspension effect and to the lack of equilibrium between the soil and aqueous phases, are discussed. Numerical values of some errors are given. The suggested cell for pH measurements of soil suspensions is: calomel electrode | KCl sat. | dialysate | soil suspension | glass electrode. A suitable electrode arrangement is illustrated and its applications are described. Generally the accuracy of pH measurements is $\gtrsim \pm 0{\cdot}1$ unit. O. M. WHITTON

1072. **Determination of water in soils by an indirect conductivity method.** C. K. Hancock and C. M. Hudgins, jun. (*Anal. Chem.*, 1954, **26** [11], 1738–1740).—A conductivity method for the determination of water in materials that may contain electrolytes is described. The basis of the method is extraction of the sample with a mixed organic solvent in the presence of excess of NaCl to mask the effect of contaminating electrolytes and then conductivity measurement of the NaCl-saturated extract. The best organic solvent mixture is ethanol - acetone and studies with various compositions show that conductivity is nearly linear with water contents in the range 0 to 10 per cent.
G. P. COOK

1073. **Determination of calcium carbonate in soils.** J. C. van Wesemael (*Chem. Weekbl.*, 1955, **51** [3], 35–36).—The method depends on the expulsion of air from a conical flask by the CO_2 liberated when HCl is added to a weighed amount of the soil. The loss in weight is a measure of the amount of $CaCO_3$ present. A. J. MEE

1074. **The determination of exchangeable calcium and magnesium in carbonate soils.** B. M. Tucker (*Aust. J. Agric. Res.*, 1954, **5** [4], 706–715).—The solubilities of calcium and magnesium from calcium carbonate, dolomite and magnesite in a *N* soln. of ammonium chloride in 60 per cent. ethanol (**I**), adjusted to pH 8·5 with ammonia, are not more than those in air-free water. **I** is suitable for extracting exchangeable metal cations from soil samples containing these carbonates. A single extraction yields a soln. in which Ca, Mg, Na and K may all be determined. The amounts of Ca and Mg extracted by this soln. from soil samples presumed to be in equilibrium with, but not containing, carbonates, are not altered by adding an excess of $CaCO_3$ or dolomite. An examination of 18 carbonate soils gave mean values of 16·5 milli-equiv. of Ca. and 11·7 of Mg per 100 g of sample compared with 16·4 and 11·7 milli-equiv., respectively, by a double-leaching method with *N* NaCl solution.
O. M. WHITTON

See also Abstract 995, 1094.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1075. **An apparatus for continuous electrophoresis on paper.** E. S. Holdsworth (*Biochem. J.*, 1955, **59** [2], 340–345).—A flowing-electrode system enables the apparatus described to be used for long periods without change of pH on the paper. Arrangements for cooling make it applicable to thermolabile substances; cooling also gives more reproducible results because evaporation of the solvent from the electrolyte is prevented. The apparatus has been used for continuous separation of mixtures in amounts up to 4 ml of 5 per cent. solutions per 24 hr. Sharp resolution is attained when the supporting medium is a thin sheet of close-textured paper; a little sharpness is lost by addition of the cooling system. J. N. ASHLEY

1076. **Automatic micro-extraction apparatus.** M. Branica (*Arhiv Kem., Zagreb*, 1954, **26** [2], 119–120).—An apparatus for the extraction of solutes from solutions by means of immiscible solvents of densities different from those of the solutions is described and illustrated. R. TRUSCOE

1077. **Paper fraction analyser.** N. Tamiya, Y. Yoshino and T. Soda (*Bull. Chem. Soc. Japan*, 1954, **27** [6], 393–394).—The separation of Hg^{++}, Cd^{++} and Cu^{++} can be achieved with a simple paper fraction analyser, individual drops which are collected on a moving paper strip, issue from a 5·1-cm ion-exchange column of Amberlite IR-120, through which is passed a 0·033 *M* soln. of their sulphates in 0·31 *N* HNO_3. The paper is subsequently sprayed with $(NH_4)_2S$ soln., then immersed in paraffin and the metal sulphides are estimated colorimetrically. Similarly, alanine, lysine, histidine and arginine may be separated on a 5·0-cm column of Amberlite XE-64 with a buffer soln. of pH 5·95 (a mixture of 2 pt. of 0·2 *M* Na citrate soln. and 1 pt. of 0·2 *M* Na phosphate soln.). G. R. WHALLEY

1078. **Universal laboratory apparatus for fractional distillation.** C. Capitani and E. Milani (*Chim. e Ind.*, 1954, **36** [9], 671–684).—Flexible standard interchangeable laboratory apparatus with a throughput of 200 to 4000 ml per hr., a hold-up of 2·5 to 45 ml and a resolving power of 4 to 24 theoretical plates is described. R. TRUSCOE

1079. **A distillation flask safe against spurting.** A. Dobrowsky (*Mitt. Chem. Forsch. Inst. Öst.*, 1954, **8** [5], 126–127).—The distillation flask described prevents boiling-over of the distilling liquid and also bumping. The flasks can be constructed in all sizes. R. J. MAGEE

1080. **A modified Conway unit for micro-diffusion analysis.** K. J. Öbrink (*Biochem. J.*, 1955, **59** [1], 134–136).—The Conway unit for micro-diffusion analysis is modified by the addition of a peripheral chamber, which is filled with the same liquid as the outer diffusion chamber (except for the fluid to be analysed). An inverted Petri dish dipping into the peripheral chamber forms a liquid trap; this avoids absorption of Br or Cl by the fixative. The apparatus is more suitable for use when relatively high temp. are required. Gas leakage is impossible in the modified unit. The apparatus is more easily cleaned than those in which paraffin, Vaseline or silicones are used. J. N. ASHLEY

1081. **An absolute rheometer of the Couette type.** K. H. Grodde (*Kolloidzschr.*, 1954, **139** [1–2], 91–96).—The factors affecting the accuracy of the Couette type of viscometer are discussed. A precision instrument is described. The ratio of the external to internal diameter of the annulus is 1·05; the cord applying the torque to the inner cylinder wraps round a logarithmic cam, and the load is variable. The outer cylinder can be rotated at a number of fixed speeds. The range 1 to 20,000 centipoises can be covered with an accuracy of $\pm$ 2 per cent. A. B. DENSHAM

1082. **Two portable pumps for fume sampling.** E. A. B. Birse (*Chem. & Ind.*, 1954, [41], 1250–1252).—A portable diaphragm gas pump can be constructed from a car petrol pump by substituting thin rubber discs for the brass flap valves. The pump is driven by Ni - Fe accumulators, the max. current consumption being $\pm$ 1·75 amp. when loaded. When there is no suction load, the max. rate of pumping is 7 to 8 cu. ft. of air per hr., and with 2 Dreschel bottles in series (each tube immersed in 1½ in. of H_2O) 6 to 7 cu. ft. of air per hr. Pumping rates down to 0·5 cu. ft. of air per hr. are attained by using a screw clip on the tube connecting the pump with the meter. A double-diaphragm pump can also be constructed from a dual car petrol pump. The pumping characteristics under the same conditions differ by a factor of $\simeq$ 2, but its current consumption is only $\simeq$ 50 per cent. greater. J. H. WATON

1083. **A new type of absolute manometer.** R. G. Nester (*Rev. Sci. Instrum.*, 1954, **35** [11], 1136–1137).—The upper end of the closed limb of a U-tube absolute manometer is terminated by a Teflon needle valve. Gas bubbles, which find their way into this limb, are forced out beyond the valve, which in operation is sealed by the manometric liquid. G. SKIRROW

1084. **An instrument for leak detection and pressure measurement in high-vacuum systems.** E. G. Leger (*Canad. J. Technol.*, 1954, **32** [6], 199–205).—An instrument for measuring gas pressures in the range 10^{-1} to 10^{-7} of Hg is described; it involves the use of vacuum thermocouples at the higher pressures and ionisation gauges at the lower pressures. For the detection of leaks, oxygen is sprayed on to the outside of the vacuum system and observation is made of the decrease in electrons from a tungsten filament in an ionisation gauge, when it is operated as a saturated diode. Leaks are traced either by observing a meter or by the change in frequency of an audio-oscillator. Damage is prevented by means of an automatic cut-off of gauges and pumps, when a pre-determined pressure is reached. Leaks in an all-metal system are conveniently sealed by brushing the surface with a dipping varnish prepared from Araldite 985-E and then baking the surface in a blue bunsen flame. G. HELMS

1085. **A modified hand-operated high-pressure hydrogen sulphide generator.** P. Heath (*Analyst*, 1954, **79**, 787–788).—The H_2S generator described is simpler to construct and easier to dismantle, clean and re-assemble than the two versions previously described (Stock *et al.*, *Brit. Abstr. C*, 1951, 487; 1953, 239). By means of two valves easily constructed from glass tubing and rubber and hand-operated by a rubber teat acid is drawn into a generating funnel and the evolved gas is driven into a gas reservoir connected with a capillary jet. Manual operation of the pumping unit 3 or 4 times

a minute produces adequate pressure in the gas reservoir and a continuous flow of H_2S through the jet. A. O. JONES

1086. **Construction of a robust torsion microbalance.** I. Graham (*Mikrochim. Acta*, 1954, [6], 746–759).—The balance described carries a load of 0·5 g, which can be weighed with a standard deviation of $\simeq 10$ μg. It is robust and easy to operate. A. J. MEE

1087. **Magnetically operated balance for collection of liquid fractions of equal weight.** D. H. Simmonds and K. I. Wood (*Anal. Chem.*, 1954, **26** [11], 1860–1862).—Liquid collects in a cup until its weight counterbalances the attraction of a magnet for an iron bolt. The liquid is then discharged into a vessel. For accurate collection of small fractions the apparatus is superior to other methods of fraction dispensing. The apparatus described can be adjusted to deliver fractions in the range 0·5 to 2·0 g. A. J. MEE

1088. **Vibrationless weighing bench for microbalances.** H. Gysel and W. Strebel (*Mikrochim. Acta*, 1954, [6], 782–784).—The table for precision balances described is insensitive to vibrations. The heavy plate of the table is supported by four springs embedded in a highly viscous damping material. A. J. MEE

1089. **Density sensitive device for continuous measurement of small concentration changes.** R. M. Butler and A. C. Plewes (*Chem. Can.*, 1954, **6** [11], 52–54).—The instrument, which can detect concn. changes of 0·1 per cent., is designed for the continuous measurement of small concn. changes, *e.g.*, on a chemical plant. The solution, after being de-gassed, flows over a bob, and the temp. is adjusted in a coil so that the bob has zero buoyancy. The movement of the bob actuates a photo-electric cell, relay circuit, temp. recorder and a vacuum system, which controls the temp. of the solution by altering the pressure of the vapour surrounding the bob. For aqueous solutions, the instrument responds to density changes of 0·2 pt. per 1000. D. R. PECK

1090. **Testing of hydrometers.** J. C. Hughes (U.S. Dep. Comm. Nat. Bur. Stand. Circ. No. 555, 10 pp.).—Recommendations for the construction of different types of hydrometer are outlined, together with the definition and method of graduation of various scales. The methods of manipulation used by the Bureau of Standards to secure accuracy and eliminate errors from temp., surface tension, etc., and the tests that are carried out are described briefly. J. M. JACOBS

1091. **A portable electric velometer for low-velocity liquids.** A. D. Misener (*Canad. J. Technol.*, 1954, **32** [6], 242–249).—An electrical equipment capable of measuring liquid-flow velocities up to 4 m.p.h. with an accuracy $> 0{\cdot}1$ m.p.h. is described. The principle is the same as that used in a hot-wire anemometer, *i.e.*, the heat taken from a detector heated at a constant rate is directly related to the rate of liquid flow by the equation $V = \frac{C}{(\Delta T)^2}$, where V = flow velocity, ΔT is the temp. diff. between the heater and the flowing liquid and C is a constant. The apparatus comprises a small bead thermistor encased in a thin-walled tube, enclosed in a second thin-walled tube fitted with a heating coil; this structure is mounted in a stout brass tube, through which the electrical leads from the temp.-sensitive element and the heater are taken to terminals mounted on a handle at the top of the brass tube. The detector is connected to the battery power supply, adjusting circuits and indicating meters. The methods of calibration and operations are described, and results obtained in river waters are discussed. G. HELMS

1092. **Volume collector for chromatographic column.** P. B. Hamilton (*Anal. Chem.*, 1954, **26** [11], 1857).—A siphon apparatus for use with the Technicon drop-counting fraction collector is described. A. J. MEE

1093. **Automatic fraction collector.** D. Fraser (*Anal. Chem.*, 1954, **26** [11], 1858–1859).—The fraction collector consists of a motor-driven conveyor chain activated by an impulse from a positive direct-contact drop-counter accumulator. The individual links of the conveyor chain are the collection-tube holders. A. J. MEE

1094. **Non-contaminating nylon slip-roll pulveriser for grinding dry plant samples.** A. E. Kretschmer, jun., and J. W. Randolph (*Anal. Chem.*, 1954, **26** [11], 1862).—The apparatus consists of three roughened nylon rollers, which pulverise dry plant samples without the risk of contamination from most of the elements essential for plant growth. A. J. MEE

1095. **Crystallisation of milligram quantities of organic compounds.** G. F. Wright (*Canad. J. Technol.*, 1954, **32** [6], 250–252).—A device for (fractional) crystallisation of 1 to 100 mg of material is described, which eliminates losses by evaporation of solvent, evaporation from filter-paper, or from decolorising absorbent. It comprises a Pyrex-glass tube with side-arms which are plugged with cotton-wool, one plug serving as a safety precaution and the other as a filtering medium. Substance and solvent are placed in the tube, which is then corked, and the solute obtained is ejected through the filter side-arm by air pressure entering through the other arm. Dimensions and other details of the apparatus are given. G. HELMS

See also Abstracts 855, 889, 1021, 1029, 1052, 1055.

Optical

1096. **Adjustable bilateral slit for use with a photomultiplier in a soft X-ray spectrometer.** P. Fisher (*J. Sci. Instrum.*, 1955, **32** [1], 32–33).—A slit is described that provides a variation in width from nil to 1·5 mm, with a slit-length of 12·5 mm; adjustment is made by a micrometer head. Widths are reproducible to within 0·002 mm. G. SKIRROW

1097. **Quanta counter and spectral-analyser.** Chr. Smit (*Chem. Weekbl.*, 1954, **50** [51], 889–894).—A spectral-analyser and a quanta counter constructed in the Physical Laboratory, University of Utrecht, are described. The properties of the photomultipliers used in the instruments are discussed with special reference to their effect on sensitivity. In the determination of copper, with silver as an internal standard, the spectral-analyser enables intensity ratios to be determined with a 6-fold increase in precision over that attained with photographic plates. E. HAYES

1098. **Quantometry.** H. H. K. Rossmark (*Chem. Weekbl.*, 1954, **50** [51], 894–898).—The general principles underlying the automatic recording of the results of determinations made with emission spectra are discussed. Examples are given of direct and integrating quantometers. The use of

the Baird - Dow quantometer in the analysis of low-alloy steels is described, and several modifications in the construction of this instrument are proposed with the object of increasing its value for routine work. E. HAYES

1099. **The electric spark as an excitation source in spectrochemistry.** H. W. Deinum (*Chem. Weekbl.*, 1954, **50** [51], 881–884).—The theory of the oscillatory circuit for the production of spark discharges is discussed, and the effect of variations in capacitance, inductance, resistance and electrode gap on the spark spectra is examined. E. HAYES

1100. **Quantitative spectrochemical analysis by means of the direct-current carbon arc.** N. H. Addink (*Chem. Weekbl.*, 1954, **50** [51], 885–888).—The use of the direct-current carbon arc in quantitative spectrochemical analysis is discussed and the advantages and disadvantages of arc and spark spectra are considered. The visual determination of the intensity of spectral lines with a standard paper-density scale is described. Experimental results indicate that the standard deviation of a single line-intensity measurement is ± 5 to ± 10 per cent. E. HAYES

1101. **Direct-reading spectrochemical analysis with a rapid-scanning spectrometer.** R. K. Brehm and V. A. Fassel (*Spectrochim. Acta*, 1954, **6** [5–6], 341–372).—The optical, mechanical and electronic features of the instrument, which is adaptable to flame, d.c. arc, and spark discharges, are described in detail. The spectra are rapidly and repeatedly scanned past a single exit-slit detector assembly, and the resulting signal is monitored on an oscillograph in which the horizontal sweep is synchronised to the spectrum scan. Line intensities are evaluated by impressing the deflecting signal from the cathode-ray tube on to a separate cathode-ray tube equipped with a fast-decay phosphor and no horizontal sweep. A grid of 50 opaque lines is placed in contact with the screen of this second tube, and as the luminous spot is deflected by the spectrum signal it is alternately covered and uncovered by the opaque lines. A secondary multiplier-phototube detects the resulting light-pulses and converts them to a pulsed signal containing the amplitude information. Hence, as a spectrum-line crosses the exit-slit a "pulse-burst" is produced containing a number of pulses proportional to the instantaneous intensity of the line. The pulse-bursts corresponding to specific lines are separated by means of synchronised gates. High-speed scalers evaluate the number of pulses in the bursts, so that after an exposure the total counts are proportional to the integrated intensities of the lines. Some analytical results obtained are reported; in the determination of Ba to Ca ratios in $BaCO_3$ - $CaCO_3$ mixtures by d.c. arc excitation, the coeff. of variation was $\pm 4{\cdot}7$ per cent., whilst in determinations of 0·05 to 0·5 per cent. of Cu in SnO_2 it was ± 8 per cent. (by d.c. arc) and only $\pm 0{\cdot}58$ per cent. when the overdamped condenser discharge-conducting briquette technique was used. W. J. BAKER

1102. **Simplified line-width procedure for measuring dense spectrum lines.** F. Farrell (*Spectrochim. Acta*, 1954, **6** [5–6], 442–443).—Dense spectrum lines (photographic blackening > 2) are read by opening the slit of the microphotometer wider than the line image, traversing the line normally and recording the galvanometer reading, which, at constant slit-width, varies inversely as line-width. The latter is proportional to concn. of the element. When the Na doublet at 5890 A has wide lines, the gap between (which varies as line-width) is read. The procedure is useful for the spectrograms of permanent-magnet alloys and in the analysis of tungstic, ferric and nickel oxides. W. J. BAKER

1103. **Infra-red analysis of solids by potassium bromide pellet technique.** D. N. Ingebrigtson and A. L. Smith (*Anal. Chem.*, 1954, **26** [11], 1765–1768).—The optimum conditions for producing pellets of KBr for the infra-red analysis of solids have been investigated. For pellets of one-inch diameter, a total force of $\simeq 20$ tons applied for 20 min. gave permanently clear discs, but reasonably satisfactory results could be obtained by pressing for 3 to 5 min. at slightly higher pressures. A simple evacuable die is described in which the pellet is pressed directly into a holder which fits into the spectrometer, thus avoiding handling of the pellet. Good quality spectra can be obtained by making a slurry of the sample and KBr with a volatile solvent, such as acetone or benzene, and grinding until dry. The technique can be used for both qual. and quant. analyses. A. J. MEE

1104. **Note on the potassium bromide disc technique for measurements of infra-red spectra.** N. Clauson-Kaas, P. Nedenskov, B. Bak and J. R. Andersen (*Acta Chem. Scand.*, 1954, **8** [6], 1088–1089).—The technique of the preparation of satisfactory discs for use in the measurement of infra-red spectra is discussed. Grinding the sample with KBr followed by pressing, regrinding of the disc and pressing again gave consistently reproducible results. Details are given of the pressing tools used, the preparation of the KBr and of the discs. N. E.

1105. **Micro-wave absorption cell for reactive molecules.** W. A. Hardy, P. Fletcher and V. Suarez (*Rev. Sci. Instrum.*, 1954, **25** [11], 1135).—A Stark modulation micro-wave absorption cell is proposed for use with reactive compounds, *e.g.*, fluorides, over the temp. range $-72°$ C to $+100°$ C. Window seals are ensured by using sheet Teflon gaskets. The only metallic surfaces exposed to the gas are brass and copper. G. SKIRROW

See also Abstracts 890, 1027.

Electrical

1106. **Derivative polarography. I. A new circuit for derivative polarography.** M. Ishidate, T. Isshiki, Y. Mashiko and N. Hosoya (*Pharm. Bull., Japan.*, 1954, **2** [3], 259–262).—A new circuit is described in detail that enables derivative polarograms to be obtained with a single dropping mercury electrode. A small a.c. voltage is superimposed upon the ordinary d.c. applied voltage and the fluctuations in cell current alone are measured. When the method was applied to several cations it was found that the max. occurred at almost the same applied d.c. potentials as the half-wave potentials. H. F. W. KIRKPATRICK

1107. **An automatic conductivity bridge for chromatographic analyses.** C.-H. de Verdier and C. I. Sjöberg (*Acta Chem. Scand.*, 1954, **8** [7], 1161–1168).—A recording device for separation in chromatographic columns is based on the construction of James, Martin and Randall (*Brit. Abstr. C*, 1951, 359); certain disadvantages have been eliminated by inserting a Wagner earth and a comparison cell of the same geometrical shape as the measuring cell. Full details and drawings of the circuit are given. N. E.

See also Abstract 1053.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
critical	crit.
crystalline	cryst.
crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment	expt.
foot, feet	ft.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
maxim -um, -a	max.
melting-point	m.p.
microcurie	μC
microgram	μg
microlitre	μl
micron	μ
milliampere	mA
millicurie	mC
milligram	mg
millilitre	ml
millimetre	mm
millimicron	mμ
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
ounce	oz
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ
relative humidity	R.H.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

Volume 2. No. 4, Abstracts 823-1107 April, 1955

ANALYTICAL ABSTRACTS

A PUBLICATION OF
THE SOCIETY FOR ANALYTICAL CHEMISTRY

EDITORIAL COMMITTEE

Chairman: R. C. Chirnside. *Members:* B. S. Cooper, B. A. Ellis, D. C. Garratt, I. Macintyre, W. A. Waygood and the President and Honorary Officers

President of the Society: K. A. Williams

Hon. Secretary of the Society: N. L. Allport

Hon. Assistant Secretary of the Society: R. E. Stuckey

Hon. Treasurer of the Society: J. H. Hamence

Editor: Norman Evers, B.Sc., Ph.D., F.R.I.C.

Assistant Editor: B. J. Walby, B.Sc., A.R.I.C.

CONTENTS

	Abstract
General Analytical Chemistry	823
Inorganic Analysis	848
Organic Analysis	931
Biochemistry	
Blood, Bile, Urine, etc.	967
Drugs	1000
Food	1021
Sanitation	1051
Agriculture and Plant Biochemistry	1068
General Technique and Laboratory Apparatus	
General	1075
Optical	1096
Electrical	1106

Printed and Published for the Society for Analytical Chemistry by W. Heffer & Sons Ltd., Cambridge, England. Communications to be addressed to the Editor, Norman Evers, 20, Eastcheap, London, E.C.3. Enquiries about advertisements should be addressed to Walter Judd Ltd., 47, Gresham Street, London, E.C.2.

Entered as Second Class at New York, U.S.A., Post Office

955

y:

ract
823
848
931

967
000
021
051
068

1075
1096
1106

gland.
uiries